AMERICAN SOCIETY
for
TESTING MATERIALS

(Reg. U.S. Pat. Office)

PROCEEDINGS

VOLUME 60
1960

COMMITTEE REPORTS
TECHNICAL PAPERS

Published by the
AMERICAN SOCIETY FOR TESTING MATERIALS
1916 Race St., Philadelphia 3, Pa.

© BY AMERICAN SOCIETY FOR TESTING MATERIALS 1961
Library of Congress Catalog Card Number: 8-5854

Printed in Baltimore, U. S. A
April, 1961

FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings,* together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* include those accepted for publication in the ASTM Bulletin or in Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1960 is given on page 1241 of this volume. This supplements the lists appearing in the *Proceedings* from 1948 to 1959 covering all special publications published by the Society up to and including 1959.

Nothing contained in any publication of the American Society for Testing Materials is to be construed as granting any right, by implication or otherwise, for manufacture, sale, or use in connection with any method, apparatus, or product covered by Letters Patent, nor as insuring anyone against liability for infringement of Letters Patent.

The Society is not responsible, as a body, for the statements and opinions advanced in this publication

CONTENTS

PROCEEDINGS

PAGE
Summary of Proceedings of the Sixty-third Annual Meeting 1
Summary of Proceedings of Chicago Spring Meeting—Panel Discussion on Appearance of Aluminum Surfaces 1205
Annual Address by the President—F. L. LaQue 37
Annual Report of the Board of Directors 40
  Appendix I. Report of the Auditors for the Fiscal Year November 29, 1958 to November 30, 1959 57
  Appendix II. Report of Administrative Committee on Standards 71
  Appendix III. Report of Administrative Committee on Research 78
  Appendix IV. Report of Administrative Committee on Papers and Publications. 79
  Appendix V. Report of Administrative Committee on District Activities 84
  Appendix VI. Report of Administrative Committee on Simulated Service Testing 85
  Appendix VII. Report of Administrative Committee on Education in Materials. 86

COMMITTEE REPORTS

Ferrous Metals

Report of Committee A-1 on Steel 88
  Appendix. Proposed Recommendations Affecting Standards on Steel 93
Report of Committee A-3 on Cast Iron 108
Report of Committee A-5 on Corrosion of Iron and Steel 111
  Appendix. Field Tests of Atmospheric Corrosion of Hardware (1929 Program and 1958 Program) 116
Report of Committee A-6 on Magnetic Properties 124
Report of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys 126
  Compilation of Chemical Compositions and Rupture Strengths of Super-Strength Alloys—Ward F. Simmons and M. C. Metzger (ASTM Special Technical Publication No. 170-B

Non-Ferrous Metals

Report of Committee B-1 on Wires for Electrical Conductors 130
Report of Committee B-2 on Non-Ferrous Metals and Alloys 135
Report of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys 141
  Final Report: Measurement of Surface Moisture and Sulfur Dioxide Activity at Corrosion Sites—P. J. Sereda (See ASTM BULLETIN, *No. 246, May, 1960, p. 47)*
Report of Committee B-4 on Materials for Thermostats and for Electrical Resistance, Heating, and Contacts 143
Report of Committee B-5 on Copper and Copper Alloys, Cast and Wrought 146
  Appendix. Recommendations Affecting Standards for Copper and Copper Alloys, Cast and Wrought 149
  The Tensile Properties of Hard-Drawn Alloy A Phosphor Bronze Rod—George R. Gohn and W. G. Bader 843
Report of Committee B-6 on Die-Cast Metals and Alloys 160
  Appendix I. Mechanical Properties of Zinc Alloy Test Bars Produced in Evacuated Dies 162
  Appendix II. Exposure Tests of SC84A Aluminum Die-Cast Test Bars with Varying Zinc Contents 165
Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought 170
  Appendix I. Proposed Recommendations Affecting Standards on Light Metals and Alloys, Cast and Wrought 174

PAGE
  Appendix II. Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings—L. H. Adam and M. Dougherty 186
Report of Committee B-8 on Electrodeposited Metallic Coatings 204
  Appendix I. Report of Subcommittee III on Conformance Tests—Part I: Thickness Determination of Electrodeposits 207
  Appendix II. Report of Subcommittee III on Conformance Tests—Part II: Evaluation of Measuring Devices for Thickness of Zinc Electrodeposited on Steel 218
Report of Committee B-9 on Metal Powders and Metal Powder Products 230

Report of Advisory Committee on Corrosion 234
Report of the Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals 241

Cementitious, Ceramic, Concrete, and Masonry Materials

Report of Committee C-1 on Cement 247
Report of Committee C-3 on Chemical-Resistant Mortars 253
Report of Committee C-4 on Clay Pipe 255
Report of Committee C-7 on Lime 257
Report of Committee C-8 on Refractories 260
Report of Committee C-9 on Concrete and Concrete Aggregates 266
Report of Committee C-11 on Gypsum 272
Report of Committee C-13 on Concrete Pipe 275
Report of Committee C-15 on Manufactured Masonry Units 277
Report of Committee C-16 on Thermal Insulating Units 281
Report of Committee C-17 on Asbestos-Cement Products 284
Report of Committee C-18 on Natural Building Stones 288
Report of Committee C-20 on Acoustical Materials 289
Report of Committee C-21 on Ceramic Whitewares and Related Products 291
Report of Committee C-22 on Porcelain Enamel 295
Report of Committee C-23 on Sorptive Mineral Materials 297
Report of Committee C-24 on Joint Sealants 298

Miscellaneous Materials

Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products 300
  Appendix I. Historical Review and Supporting Data on the Methods of Test for Phthalate Ester Color of High-Gravity Glycerin 311
  Appendix II. Experimental Data on Method of Test for Urea Content of Nitrogen Resins 314
  Appendix III. Results of an Interlaboratory Study on Viscosity of Resin Solutions 315
Report of Committee D-2 on Petroleum Products and Lubricants 316
  Procedure for Determination of Tetraethyllead in Gasoline—Comparison of ASTM Method D 526 and GOST-63 (See ASTM BULLETIN, *No. 250, Dec., 1960, p. 13)*
Report of Committee D-3 on Gaseous Fuels 326
Report of Committee D-4 on Road and Paving Materials 328
Report of Committee D-5 on Coal and Coke 332
Report of Committee D-6 on Paper and Paper Products 336
Report of Committee D-7 on Wood 339
Report of Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses 345
Report of Committee D-9 on Electrical Insulating Materials 347
Report of Committee D-10 on Shipping Containers 353
Report of Committee D-11 on Rubber and Rubber-like Materials 355
Report of Committee D-12 on Soaps and Other Detergents 361
Report of Committee D-13 on Textile Materials 365
  Appendix. Recommendations Affecting Standards on Textile Materials 367
Report of Committee D-14 on Adhesives 371
Report of Committee D-15 on Engine Antifreezes 375
Report of Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials 377
  Appendix. Proposed Recommendations for Interlaboratory Testing of Industrial Aromatic Hydrocarbons and Related Materials 381

PAGE
Report of Committee D-17 on Naval Stores 386
Report of Committee D-18 on Soils for Engineering Purposes 388
Report of Committee D-19 on Industrial Water 393
Report of Committee D-20 on Plastics 402
Report of Committee D-21 on Wax Polishes and Related Materials 415
Report of Committee D-22 on Methods of Atmospheric Sampling and Analysis 417
Report of Committee D-23 on Cellulose and Cellulose Derivatives 420
Report of Committee D-24 on Carbon Black 423
Report of Committee D-25 on Casein and Similar Protein Materials 426
Report of Committee D-26 on Halogenated Organic Solvents 428
Report of Committee D-27 on Electrical Insulating Liquids and Gases 429
A More Sensitive Method for Dielectric Strength of Insulating Oil—P. F. Ast (*See* ASTM BULLETIN, *No. 250, Dec., 1960, p. 36*)
Report of ALCA-ASTM Joint Committee on Leather 435

Miscellaneous Subjects

Report of Committee E-1 on Methods of Testing 439
Rheological Testing of Elastomers at Low Temperatures—Part II—L. Boor, M. Ilanok, F. Conant, and W. Scoville, Jr. (*See* ASTM BULLETIN, *No. 246, May, 1960, p. 25*)
Report of Committee E-2 on Emission Spectroscopy 448
Report of Committee E-3 on Chemical Analysis of Metals 452
Report of Committee E-4 on Metallography 457
Report of Committee E-5 on Fire Tests of Materials and Construction 460
Appendix. Proposed Method of Test for Surface Flammability of Building Materials Using an 8-ft Tunnel Furnace 462
Report of Committee E-6 on Methods of Testing Building Constructions 468
Report of Committee E-7 on Nondestructive Testing 471
Report of Committee E-9 on Fatigue 474
Report of Committee E-10 on Radioisotopes and Radiation Effects 476
Report of Committee E-11 on Quality Control of Materials 477
Report of Committee E-12 on Appearance 480
Report of Committee E-13 on Absorption Spectroscopy 482
Report of Committee E-14 on Mass Spectrometry 484
Report of Committee E-15 on Analysis and Testing of Industrial Chemicals 486
Appendix. Proposed Glossary of Statistical Terms (Part A of Proposed Recommended Practice for Developing Precision Data on ASTM Methods for the Analysis and Testing of Industrial Chemicals) 489

Materials for Specific Applications

Report of Committee F-1 on Materials for Electron Tubes and Semiconductor Devices 491
Report of Committee F-2 on Flexible Barrier Materials 495

TECHNICAL PAPERS

Edgar Marburg Lecture

Utilization of Solar Energy—Farrington Daniels (*issued only as separate pamphlet*)

Gillett Memorial Lecture

Nuclear Fuel Element Development—B. Carson Dalzell (*issued only as separate pamphlet*)

Properties of Crystalline Solids

Summary of Proceedings of Sessions Dealing with Properties of Crystalline Solids (ASTM *Special Technical Publication No. 283*) 499

Fatigue

Fatigue Properties of Some High-Strength Steels—H. E. Frankel, Jr., J. A. Bennett and C. M. Carman 501
Discussion 509
Low-Cycle Fatigue of Pressure-Vessel Materials—G. Sachs, W. W. Gerberich, V. Weiss, and J. V. Latorre 512

PAGE

Elevated-Temperature Tensile and Fatigue Behavior of Unalloyed Arc-Cast Molybdenum—G. W. Brock and G. M. Sinclair 530
Discussion 538
New Observations Relating to the Mechanism of Fatigue Failure—Melvin A. Wilkov 540
Discussion 559
The R. R. Moore Rotating-Beam Fatigue Test—G. R. Gohn and A. Fox 560
Discussion 568
Additional Studies of Effects of Anodic Coatings on the Fatigue Strength of Aluminum Alloys—G. W. Stickley 577
Discussion 588
Design Applications for Improving Fatigue Resistance of Airplane Structures—C. R. Smith 589
Discussion 601
The Fatigue Notch Sensitivity of Annealed Copper—J. Y. Mann 602
Static and Fatigue Strength of Metals Subjected to Triaxial Stresses—William M. Lehrer and Harry Schwartzbart 610
Relation Between Rolling-Contact Fatigue Life and Mechanical Properties for Several Aircraft Bearing Steels—E. V. Zaretsky and W. J. Anderson 627
Discussion 643

Metals

Long-Time Atmospheric Corrosion Tests on Low-Alloy Steels—H. R. Copson 650
Discussion 666
Effects of Alloying Additions on Hot Cracking of Austenitic Chromium-Nickel Stainless Steels—Frederick C. Hull 667
Discussion 686
Carbon:Strength Relationships in 5 per cent Chromium Ultra-High-Strength Steels—John C. Hamaker, Jr. and Eugene J. Vater 691
Discussion 717
Spin-Fracture Tests on Nickel-Molybdenum-Vanadium Rotor Steels in the Brittle Fracture Range—G. O. Sankey 721
Discussion 733
Temperature Dependence of the Elastic Moduli of Several Stainless Steels—F. Garofalo 738
Delayed Failure of High-Strength Steel in Liquid Environments—E. A. Steigerwald 750
Effect of Carbon Content and Melting Practice on Room-Temperature Sharp-Edge-Notch Tensile Characteristics of H-11 Modified and 300M Sheet Steels—J. L. Shannon, Jr., G. B. Espey, A. J. Repko, and W. F. Brown, Jr. 761
Discussion 778
Mechanical and Physical Properties of Five Copper-Base Casting Alloys—W. H. Johnson and J. G. Kura 796
Discussion 811
Structural Beryllium—B. B. Muvdi 812
Discussion 823
A Study of Mechanical Behavior of Inconel 718 Alloy for Design of a Glide Vehicle—N. A. Tiner and J. E. Fischler 825
Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings—L. H. Adam and M. Dougherty (Appended to the B-7 Report, p. 186)
Report on Twenty-Year Atmospheric Corrosion of Zinc-Coated and Uncoated Wire and Wire Products—Fred M. Reinhart (ASTM Special Technical Publication No. 290)
The Tensile Properties of Hard-Drawn Alloy A Phosphor Bronze Rod—G. R. Gohn and W. G. Bader 843

Papers on High Temperature

The Role of Refractory Metals in Superalloys—Earl R. Parker 849
Discussion 866
Relaxation Characteristics of Inconel at Elevated Temperatures—C. R. Kennedy and D. A. Douglas 867
Discussion 881
Creep-Rupture Properties of a Carbon Steel and a Low-Alloy Steel at 1200 to 1800 F—P. N. Randall 885
Discussion 893
Predicting Creep Deflections of Plastic Beams—Charles A. Schulte 895

PAGE

An Examination of High-Temperature Stress-Rupture Correlating Parameters—Francis J. Clauss 905
Strength of Structural Alloys Under Rapid Heating and Loading—J. R. Kattus and D. W. McDowell, Jr. 928
Compression Testing of Sheet Magnesium Utilizing Rapid Heating—Raymond W. Fenn, Jr. 940
  Discussion 955
Preliminary Evaluation of Thermocouple Attachment Methods for Radiant-Heating Applications—Jay F. Burcham 957
  Discussion 971
Application of Induction Heating to Short-Time Elevated-Temperature Tension Testing—Albert P. Levitt and Anthony G. Martin 974
  Discussion 984
The Phenomenon of Stress-Relaxation—A. M. Freudenthal 986
  Discussion 1000
Report on the Elevated-Temperature Properties of Aluminum and Magnesium Alloys—Howard R. Voorhees and James W. Freeman (ASTM Special Technical Publication No. 291)

Symposium on Acoustical Fatigue

Summary of Proceedings of the Symposium on Acoustical Fatigue (*ASTM Special Technical Publication No. 284*) 1001

Symposium on Shear and Torsion Testing

Summary of Proceedings of the Symposium on Shear and Torsion Testing (*ASTM Special Technical Publication No. 289*) 1002

Symposium on Methods of Metallographic Specimen Preparation

Summary of Proceedings of the Symposium on Methods of Metallographic Specimen Preparation (*ASTM Special Technical Publication No. 285*) 1003

Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials

Summary of Proceedings of the Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials (*ASTM Special Technical Publication No. 287*) 1004

Concrete, Cement, and Plaster

The Reproducibility of Flexure and Compression Tests for Determining the Strength of Cement—M. F. Kaplan 1006
  Discussion 1018
An X-ray Diffraction Investigation of Hydrated Portland-Cement Pastes—D. L. Kantro, L. E. Copeland, and Elaine R. Anderson 1020
  Discussion 1035
Investigation of the Fine and Coarse Aggregates of Northern Illinois for Alkali Reactivity—J. F. Weigel 1036
Performance Tests of Field Concrete Mixers—I. Narrow 1047
  Discussion 1061
Influence of Physical Characteristics of Aggregates on Frost Resistance of Concrete—George Verbeck and Robert Landgren 1063
The Effects of Early Freezing on Low-Density Aggregate-Type Concrete—I. A. Benjamin and G. D. Ratliff, Jr. 1080
Effect of High Temperatures on Concretes Incorporating Different Aggregates—N. G. Zoldners 1087

Soils

Observed Settlement of Preloaded Soft, Fine-Grained Soils—E. J. Zegarra 1109
  Discussion 1118
The Compressive Strength of Remolded Niagara Clay—D. J. Bazett and S. W. Smotrych 1124
  Discussion 1132
Duplication of Prototype Stress-Strain Relations in Soil Masses by Laboratory Tests—Richard G. Ahlvin and Donald N. Brown 1137
  Discussion 1149
The Importance of Freezing Rate in Frost Action in Soils—E. Penner 1151

PAGE

Symposium on Nuclear Methods for Measuring Soil Density and Moisture

Summary of Proceedings of the Symposium on Nuclear Methods for Measuring Soil Density and Moisture (*ASTM Special Technical Publication No. 293*) 1166

Papers on Road and Paving Materials

Summary of Proceedings of the Session on Road and Paving Materials (*ASTM Special Technical Publication No. 294*) 1167

General Testing

Flow Properties of Bulk Solids—A. W. Jenike, P. J. Elsey, and R. H. Woolley 1168
Discussion 1182
A Notched Ring Specimen for Hydrogen Embrittlement Studies—F. S. Williams, W. Beck, and E. J. Jankowsky 1192
Discussion 1199
Strength and Related Properties of Wood Poles—Final Report—Lyman W. Wood, E. C. O. Erickson, and A. W. Dohr (ASTM Special Technical Publication No. 295)

Symposium on Radiation Effects and Radiation Dosimetry

Summary of Proceedings of the Symposium on Radiation Effects and Radiation Dosimetry (*ASTM Special Technical Publication No. 286*) 1203

Symposium on Quality of Observations

Summary of Proceedings of the Symposium on Quality of Observations (*Materials Research & Standards, Vol. 1, No. 4, April, 1961*) 1204

Panel Discussion on Appearance of Aluminum Surfaces

Summary of Proceedings of the Panel Discussion on Appearance of Aluminum Surfaces 1205
List of Technical Papers Published in ASTM BULLETIN 1206
Author Index 1209
Subject Index 1224
List of Special Technical Publications 1241

SUMMARY OF PROCEEDINGS

OF THE

SIXTY-THIRD ANNUAL MEETING

ATLANTIC CITY, N. J., JUNE 26–JULY 1, 1960

This summary of the Sixty-third Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 26–July 1, 1960, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 37 technical sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 1858; committee members, 1106; guests, 375; total, 3339; ladies, 449.

In addition, a meeting of Committee E-14 on Mass Spectrometry was held in conjunction with the Annual Meeting but with separate registration at the Hotel Dennis. It had an attendance of 355.

The Proceedings are set forth session by session. There were 65 reports and 117 formal papers presented. The record with respect to each has been briefed; the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in," it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in" are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

All papers, unless otherwise noted, are published in the *Proceedings*.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded.

---

[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, will be canvassed on September 19, 1960. See Editor's Note, p. 35.

LOCATION OF ACTION ON COMMITTEE REPORTS

PAGE

Committee:

- Board of Directors 8
- A-1 on Steel 17
- A-3 on Cast Iron 26
- A-5 on Corrosion of Iron and Steel 26
- A-6 on Magnetic Properties 9
- A-7 on Malleable Iron Castings 9
- A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys 9

- B-1 on Wires for Electrical Conductors 10
- B-2 on Non-Ferrous Metals and Alloys 19
- B-3 on Corrosion of Non-Ferrous Metals and Alloys 4
- B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts 10
- B-5 on Copper and Copper Alloys, Cast and Wrought 19
- B-6 on Die-Cast Metals and Alloys 19
- B-7 on Light Metals and Alloys, Cast and Wrought 33
- B-8 on Electrodeposited Metallic Coatings 10
- B-9 on Metal Powders and Metal Powder Products 10

- C-1 on Cement 34
- C-3 on Chemical-Resistant Mortars 25
- C-4 on Clay Pipe 11
- C-7 on Lime 20
- C-8 on Refractories 11
- C-9 on Concrete and Concrete Aggregates 20
- C-11 on Gypsum 34
- C-13 on Concrete Pipe 25
- C-15 on Manufactured and Masonry Units 34
- C-16 on Thermal Insulating Materials 25
- C-17 on Asbestos-Cement Products 11
- C-18 on Natural Building Stones 4
- C-20 on Acoustical Materials 4
- C-21 on Ceramic Whiteware and Related Products 21
- C-22 on Porcelain Enamel 25
- C-24 on Joint Sealants 11

- D-1 on Paint, Varnish, Lacquer, and Related Products 21
- D-2 on Petroleum Products and Lubricants 29
- D-3 on Gaseous Fuels 4
- D-4 on Road and Paving Materials 30
- D-5 on Coal and Coke 27
- D-6 on Paper and Paper Products 27
- D-7 on Wood 14
- D-8 on Bituminous Waterproofing and Roofing Materials 14
- D-9 on Electrical Insulating Materials 22
- D-10 on Shipping Containers 15
- D-11 on Rubber and Rubber-like Materials 31
- D-12 on Soaps and Other Detergents 15
- D-13 on Textile Materials 15
- D-14 on Adhesives 16
- D-15 on Engine Antifreezes 16
- D-16 on Industrial Aromatic Hydrocarbons and Related Materials 31
- D-17 on Naval Stores 22
- D-18 on Soils for Engineering Purposes 22
- D-19 on Industrial Water 32
- D-20 on Plastics 32
- D-21 on Wax Polishes and Related Materials 16
- D-22 on Methods of Atmospheric Sampling and Analysis 16

D-23 on Cellulose and Cellulose Derivatives	27
D-24 on Carbon Black	11
D-25 on Casein and Similar Protein Materials	27
D-26 on Halogenated Organic Solvents	27
D-27 on Electrical Insulating Liquids and Gases	33
E-1 on Methods of Testing	25
E-2 on Emission Spectroscopy	35
E-3 on Chemical Analysis of Metals	19
E-4 on Metallography	16
E-5 on Fire Tests of Materials and Construction	22
E-6 on Methods of Testing Building Constructions	26
E-7 on Nondestructive Testing	35
E-10 on Radioisotopes and Radiation Effects	27
E-11 on Quality Control of Materials	33
E-12 on Appearance	28
E-13 on Absorption Spectroscopy	20
E-14 on Mass Spectrometry	26
E-15 on Analysis and Testing of Industrial Chemicals	35
F-1 on Materials for Electron Tubes and Semiconductor Devices	22
Joint Committee on Effect of Temperature on the Properties of Metals	8
Joint Committee on Leather	33

FIRST SESSION—OPENING SESSION

MONDAY, JUNE 27, 9:30 A.M.

FORMAL OPENING OF THE SIXTY-THIRD ANNUAL MEETING, PRESIDENT F. L. LAQUE

The session was formally opened by President F. L. LaQue, who called attention to the very complete program of events that were to follow during the ensuing week. The meeting was then turned over to the session chairman, I. V. Williams, Bell Telephone Laboratories, Inc.

SYMPOSIUM ON RECENT PROGRESS IN MATERIALS SCIENCES[2]

SESSION CHAIRMAN: I. V. WILLIAMS

Accomplishments and Limitations of Solid State Theory—Harvey Brooks, presented by the author.

The Influence of Surfaces on the Properties of Materials—M. J. Sinnott, presented by the author.

Mechanical Properties of Semiconductors—J. N. Hobstetter, presented by the author.

Status of Ductile Ceramic Research—E. R. Parker, presented by the author.

MATERIALS SCIENCES LUNCHEON

MONDAY, JUNE 27, 12:00 NOON

TOASTMASTER: PRESIDENT F. L. LAQUE

The guest speaker was W. O. Baker, Vice-President—Research, Bell Telephone Laboratories, Inc., whose subject was "The National Role of Materials Research and Development."[3] The attendance at the luncheon was about 190.

---

[2] The Symposium on Recent Progress in Materials Sciences and the Symposium on Nature and Origin of Strength of Materials will be published as Properties of Crystalline Solids, *ASTM STP No. 283*.

[3] To be included in Properties of Crystalline Solids, *ASTM STP No. 283*.

SECOND SESSION—SYMPOSIUM ON NATURE AND ORIGIN OF STRENGTH OF MATERIALS[2]

MONDAY, JUNE 27, 2:30 P.M.

SESSION CO-CHAIRMEN: VICE-PRESIDENT A. A. BATES
J. D. SULLIVAN

Dislocation Motions and the Yield Strengths of Solids—J. J. Gilman, presented by the author.

Fatigue Strength of Crystalline Solids—G. M. Sinclair, presented by the author.

Resistance to Creep Deformation and Fracture in Metals and Alloys—Frank Garofalo, presented by the author.

Brittle Fracture and the Strength of Metals—E. T. Wessel, presented by the author.

Size and Shape Effects on Fracture of Solids—George Irwin, presented by the author.

THIRD SESSION—COMMITTEE REPORT SESSION

MONDAY, JUNE 27, 4:30 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT M. N. CLAIR

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented in the absence of the chairman by W. H. Ailor, and accepted as a report of progress.

Committee C-18 on Natural Building Stones:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and accepted as a report of progress.

Committee C-20 on Acoustical Materials:

Report presented in the absence of the chairman by W. A. Jack, and the following action taken:

Accepted as Tentative, Revisions in:

Test for Sound Absorption of Acoustical Materials in Reverberation Rooms (C 423 – 58 T)

Committee D-3 on Gaseous Fuels:

Report presented in the absence of the chairman by K. R. Knapp, secretary, and accepted as a report of progress.

FOURTH SESSION—SYMPOSIUM ON NUCLEAR METHODS FOR MEASURING SOIL DENSITY AND MOISTURE[4]

MONDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: BONNER COFFMAN

A brief digest of the papers listed below was presented by Mr. Bonner Coffman, the session chairman, after which the subject was opened for discussion with the authors participating as panel members.

Design and Application of the Nuclear Chicago Density-Moisture Gage—O. K. Neville and T. W. Van Zelst.

Design and Calibration of a Neutron Moisture Meter—K. N. Burn.

Application of Nuclear Soil Meters to Compaction Control for Airfield Pavement Construction—P. F. Carlton.

Experiences with Nuclear Moisture and Density Surface Probes on O'Hare Field Project—J. P. Gnaedinger.

Comparison of Nuclear and Sand Cone Methods of Density and Moisture Determinations for Four New York State Soils—Sidney Mintzer.

Evaluation of Nuclear Moisture-Density Testing Equipment—W. N. Carey, Jr., J. F. Shook, and J. F. Reynolds.

Panel Consultant—Abraham Schwebel.

---

[4] To be issued as *ASTM STP No. 293.*

FIFTH SESSION—CONCRETE

MONDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: J. E. GRAY

Sanford E. Thompson Award:

The Sixteenth Sanford E. Thompson Award was made to Stanton Walker, Director, and Delmar Bloem, Associate Director of Engineering, respectively, National Sand and Gravel Assn., and National Ready Mixed Concrete Assn., Washington, D. C., for their paper on "Variations in Portland Cement," published in the 1958 ASTM *Proceedings*.

Papers:

The Effects of Early Freezing on Low-Density Aggregate-Type Concrete—I. A. Benjamin and G. D. Ratliff, Jr., presented by Mr. Benjamin.

Evaluating the Performance of Stationary Concrete Mixers—Glenway Maxon, presented by the author.

Investigation of Alkali Reactivity of the Fine and Coarse Aggregates of Northern Illinois—J. F. Weigel, presented by the author.

Performance Tests of Field Concrete Mixers—I. Narrow, presented by the author.

Influence of Physical Characteristics of Aggregates on Durability of Concrete—George Verbeck and Robert Landgren, presented by Mr. Verbeck.

SIXTH SESSION—HIGH TEMPERATURE

MONDAY, JUNE 27, 8:00 P.M.

SESSION CHAIRMAN: J. J. KANTER

The Role of Refractory Metals in Superalloys—E. R. Parker, presented by the author.

The Relaxation Characteristics of Inconel at Elevated Temperatures—C. R. Kennedy and D. A. Douglas, presented by Mr. Kennedy.

Creep-Rupture Properties of a Carbon Steel and a Low-Alloy Steel at 1200 to 1800 F—P. N. Randall, presented by the author.

Predicting Creep Deflections of Beams—C. A. Schulte, presented by title only.

An Improved Microhardness Tester for High-Temperature Use—J. H. Westbrook, presented by the author.[5]

An Examination of High-Temperature Stress-Rupture Correlating Parameters—F. J. Clauss, presented by the author.

---

[5] Published in the ASTM BULLETIN, No. 246, May, 1960, p. 53.

(*Continued in Eleventh Session*)

SEVENTH SESSION—SYMPOSIUM ON ACOUSTICAL FATIGUE[6]

TUESDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: B. J. LAZAN

Material Properties Which Affect Acoustical Fatigue Life and the Role of Damping—W. J. Trapp and B. J. Lazan, presented by Mr. Lazan.

Prediction of Acoustic Fatigue Life—H. C. Schjeldrup, presented by the author.

Fatigue of Structural Metals Under Random Loading—A. M. Freudenthal, presented by the author.

The Fatigue Damaging Effect of a Random Load—Waloddi Weibull, presented by Mr. Freudenthal.

Experimental Techniques and Equipment for Acoustical Fatigue Research and Development—D. M. Forney, Jr., presented by the author.

---

[6] To be issued as *ASTM STP No. 284*.

EIGHTH SESSION—SYMPOSIUM ON SHEAR AND TORSION TESTING[7]

TUESDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: R. W. FENN

Maximum Shear Strain Measurements and True Yield Point Determination by the Use of the Photoelastic Coating Technique—Felix Zandman, presented by the author.

Stress Distribution in Single-Shear Sheet-Specimens—C. S. Yen, presented by the author.

The Design and Development of a Tensile Loaded Shear Specimen—E. J. Zapel, presented by the author.

A Preliminary Investigation into the Load Transfer Characteristics of Spot Welds Using Photoelastic Coating Techniques—N. A. Freytag, presented by the author.

Bonded Metal-to-Metal Shear Testing—L. R. Lunsford, presented by the author.

On the Lap Joint Shear Test for Metal to Metal Bonds—R. S. Shane, presented by the author.

Preparation of Metal Surfaces for Adhesive Bonding—R. S. Shane, presented by the author.

(*Continued in Twelfth Session*)

---

[7] Issued as *ASTM STP No. 289.*

NINTH SESSION—SYMPOSIUM ON METHODS OF METALLOGRAPHIC SPECIMEN PREPARATION[8]

TUESDAY, JUNE 28, 9:00 A.M.

SESSION CHAIRMAN: G. A. ELLINGER

Introduction by Mary R. Norton.

Mechanical Polishing of Metallographic Specimens—H. S. Link, presented by the author.

Use of Diamond Abrasives in Metallographic Problems—E. C. Olden, presented by the author.

Present Methods of Metallographic Specimen Preparation for Retention and Identification of Inclusions in Steel—C. G. Brandenburg, presented by the author.

Simplified Metallographic Techniques for Nuclear Reactor Materials—F. M. Cain, Jr., presented by the author.

(*Continued in Thirteenth Session*)

---

[8] To be issued as *ASTM STP No. 285.*

TECHNICAL SESSION ON ELECTRON METALLOGRAPHY

TUESDAY, JUNE 28, 9:30 A.M.

The following papers were presented at a session sponsored by Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography:

Transmission Electron Microscopy of Type 410 Stainless Steels—B. R. Banerjee.

A Technique for Removing Plastic Backing Films—T. F. Beals and W. C. Bigelow.

Direct Observations on the Structure of Ultramicrotome Metals and Alloys—V. A. Phillips.

Electron Probe Microanalysis: Technique for Calculating Nonlinear X-ray Intensities—L. S. Birks.

Diffusion Measurement in Iron-Nickel with the Electron Microanalyzer—S. Ohh and K. G. Carroll.

A Method for Applying Internal Standards in Selected Area Diffraction—G. N. Maniar and W. C. Bigelow.

A Comparator for Measuring Electron Diffraction Patterns—L. M. Niebylski, G. Poehlman, and W. C. Bigelow.

TENTH SESSION—LUNCHEON, PRESIDENT'S ADDRESS, INTRODUCTION OF NEW OFFICERS, RECOGNITION OF 40-YEAR AND 50-YEAR MEMBERS, HONORARY MEMBERSHIP, REPORT OF BOARD OF DIRECTORS, AWARDS

TUESDAY, JUNE 28, 12:00 NOON

SESSION CHAIRMAN: PAST-PRESIDENT K. B. WOODS

About 408 were present at this annual luncheon session, including a number of ladies and visitors.

Awards of Merit:

The Chairman of the 1960 Award of Merit Committee, A. A. Jones, read brief citations and presented the following to President F. L. LaQue, who conferred on them, on behalf of the Board of Directors, the Certificate of Award of Merit:

Leslie H. Adam
Frank M. Clark
Harold F. Clemmer
O. B. Ellis
Charles M. Gambrill
George R. Gohn
William N. Harrison
*Milton E. Holmberg
*Lester E. Kuentzel
Stuart M. Phelps
Harry C. Plummer
Orin W. Rees
William A. Reich
Charles H. Scholer
Walter S. Scott
*Louis Tanner
*Robert R. Thurston
Stanton Umbreit
James S. Vanick

---

* Presented in absentia.

Election of Officers:

The results of the letter ballot on election of new officers were reported by Tinius Olsen on behalf of the Teller's Committee consisting of Mr. Olsen, Chairman, and Ralph E. Green. The results were as follows:

For President, to serve for one year:
A. A. Bates, 2265 votes.
For Vice-President, to serve for two years:
R. W. Seniff, 2243 votes.
For Directors, to serve for three years:
A. R. Belyea, 2204 votes.
L. V. Cooper, 2211 votes.
J. C. Harris, 2222 votes.
J. J. Kanter, 2230 votes.
R. F. Legget, 2202 votes.
W. R. Willets, 2198 votes.

The newly-elected members of the Board of Directors were introduced, as were President-elect A. A. Bates and Vice-President-elect Seniff, and M. N. Clair, whose term as Vice-President continues another year.

Passing of the Gavel:

The gavel of authority was presented to President-elect Bates by President LaQue although the actual shift in authority does not take place until the close of the Annual Meeting. Past-President Woods then presented to Mr. LaQue a certificate, pin, and gavel for his year of service to the Society as its President.

Recognition of Retiring Past-President and Directors:

President LaQue presented framed certificates to those retiring from the Board of Directors. Mention was made that this meeting brings to a close for R. A. Schatzel nine years on the Board of Directors, as Director, Vice-President, President, and Past-President. Recognition was also given to C. L. Clark, A. E. Juve, J. H. Koenig, and R. E. Peterson as retiring Directors.

Award of Honorary Membership:

The Associate Executive Secretary read the citation and then presented to the President L. J. Markwardt who had been elected by the Board of Directors to Honorary Membership in the Society. The President then presented to him the certificate of Honorary Membership.

Frank E. Richart Award:

The Third Frank E. Richart Award was made to R. E. Davis, Director Emeritus, Engineering Materials Laboratory, University of California, who has made notable contributions to the work of the Society in the field of concrete and concrete aggregates. Mr. C. H. Scholer, Chairman of the Award Committee, read the citation and Mr. Milos Polivka, University of California, received the award for Mr. Davis.

Presidential Address:

The annual President's Address was presented by F. L. LaQue on the subject "International Aspects of ASTM Activities."[9]

Report of the Board of Directors:

R. E. Hess, Associate Executive Secretary, presented a brief résumé of the Report of the Board of Directors.

Recognition of 40-Year and 50-Year Members:

Chairman Woods called upon President LaQue to present certificates to the seven 50-year personal members, namely, C. V. Bacon, P. H. Bates, A. T. Goldbeck, A. E. Horn, W. H. Klein, M. E. McDonnell, and G. H. Woodroffe, and Mr. Goldbeck responded for this group. Chairman Woods read the names of the two companies who had been continuously affiliated with the Society for 50 years and then read the names of the 55 members, both individual and company, who had been continuously affiliated with the Society for 40 years, and certificates to that effect were presented.

---

[9] See p. 37.

ELEVENTH SESSION—HIGH TEMPERATURE

(*Continued from the Sixth Session*)

TUESDAY, JUNE 28, 2:30 P.M.

SESSION CHAIRMAN: G. V. SMITH

Strength of Structural Alloys Under Rapid Heating and Loading—J. R. Kattus and D. W. McDowell, presented by Mr. Kattus.

Compression Testing of Sheet Utilizing Rapid Heating—R. W. Fenn, presented by the author.

Preliminary Evaluation of Thermocouple Attachment Methods for Radiant Heating Applications—J. F. Burcham, presented by the author.

Application of Induction Heating to Short-Time Elevated Temperature Tensile Testing—A. P. Levitt and A. G. Martin, presented by Mr Levitt.

The Evaluation of Resistance Strain Gages at Elevated Temperatures—R. L. Bloss, presented by the author.[10]

The Phenomenon of Stress Relaxation—A. M. Freudenthal, presented by the author.

Report of Joint Committee on Effect of Temperature on the Properties of Metals—J. J. Kanter, presented by Mr. Kanter.

---

[10] Published in *Materials Research & Standards* (formerly ASTM BULLETIN), Vol. 1, No. 1, p. 9, Jan., 1961.

TWELFTH SESSION—SYMPOSIUM ON SHEAR AND TORSION TESTING[7]

(*Continued from the Eighth Session*)

TUESDAY, JUNE 28, 2:30 P.M.

SESSION CHAIRMAN: R. B. CLAPPER

Shear and Torsion Tests Used at U. S. Forest Products Laboratory for Wood, Plywood, and Sandwich Constructions—E. W. Kuenzi and W. G. Youngquist, presented by Mr. Youngquist.

The Study of High Polymers by Means of the

Torsion Pendulum—N. G. McCrum, presented by the author.

A Technique for Torsional Testing of Refractory Materials at Temperatures up to 2800 C—C. E. Waller and M. L. Stehsel, presented by Mr. Waller.

A Critical Discussion of Some Methods of Shear and Torsion Testing of Solid Materials—R. B. Clapper, presented by the author.

THIRTEENTH SESSION—SYMPOSIUM ON METHODS OF METALLOGRAPHIC SPECIMEN PREPARATION[8]

(*Continued from the Ninth Session*)

TUESDAY, JUNE 28, 2:30 P.M.

SESSION CHAIRMAN: L. L. WYMAN

The Automatic Polishing of Metallographic Specimens—R. L. Anderson, presented by the author.

The Use of Vibratory Polishing for Metallographic Sample Preparation—G. R. Grieger, presented by the author.

Application of Vibratory Polishing in Hot Cell Metallography—E. J. Long, Jr., and R. J. Gray, presented by Mr. Long.

Techniques for Controlling Surface Pitting in Polishing Ceramic Materials—R. B. Snow, presented by the author.

FOURTEENTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 28, 4:30 P.M.

SESSION CHAIRMAN: J. H. FOOTE

Committee A-6 on Magnetic Properties:

Report presented by A. C. Beiler, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Permeability of Feebly Magnetic Materials (A 342 - 53) (Standard reverted to tentative)

Test for Alternating Current Core Loss and Permeability of Magnetic Materials (A 343 - 54) (Standard reverted to tentative)

Test for Electrical and Magnetic Properties of Magnetic Materials (A 344 - 52) (Standard reverted to tentative)

Committee A-7 on Malleable-Iron Castings:

Report presented in the absence of the chairman by J. H. Lansing, secretary, and accepted as a report of progress.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented in the absence of the chairman by R. B. Gunia, and the following actions taken subject to review by the Administrative Committee on Standards of the results of the vote in Committee A-10 since the vote was not available at the time of the session:

Accepted as Tentative, Revisions in:

Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 59 T) (Jointly with Committee A-1)

Spec. for Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 - 59) (Standard reverted to tentative)

Spec. for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 55) (Standard reverted to tentative)

Spec. for Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 - 55) (Standard reverted to tentative)

Adopted as Standard, Revisions in:

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 58) (Jointly with Committee A-1)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 59) (Jointly with Committee A-1)

Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 58) (Jointly with Committee A-1)

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 59) (Jointly with Committee A-1)

Committee B-1 on Wires for Electrical Conductors:

Report presented in the absence of the chairman by C. E. Topping, Vice-Chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Nickel-Coated Soft or Annealed Copper Wire (B 355 – 60 T)
Def. of Terms Relating to Uninsulated Metallic Electrical Conductors (B 354 – 60 T)

Accepted as Tentative, Revisions in:

Spec. for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR) (B 232 – 59 T)
Spec. for Copper Conductors for Use in Hookup Wire for Electronic Equipment (B 286 – 59 T)
Spec. for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (B 341 – 59 T)
Test for Electrical Conductivity by Use of Eddy Currents (B 342 – 59 T)

Adopted as Standard:

Test for Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication (B 279 – 53 T)
Spec. for Silver-Coated Soft or Annealed Copper Wire (B 298 – 56 T)
Spec. for Aluminum Wire for Communication Cable (B 314 – 57 T)
Spec. for Half-Hard Aluminum Wire for Electrical Purposes (B 323 – 58 T)
Spec. for Rectangular and Square Bare Aluminum Wire for Electrical Conductors (B 324 – 58 T)

Adopted as Standard, Revisions in:

Spec. for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 59)
Test for Resistivity of Electrical Conductor Materials (B 193 – 59)
Spec. for Hard-Drawn Aluminum Wire for Electrical Purposes (B 230 – 58)
Spec. for Concentric-Lay-Stranded Aluminum Conductors, Hard, Three-Quarter Hard, and Half-Hard (B 231 – 59)
Spec. for Rolled Aluminum Rods (EC Grade) for Electrical Purposes (B 233 – 55)
Spec. for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 – 59)
Spec. for Zinc-Coated (Galvanized) Steel Core Wire (with Coatings Heavier than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 – 59)

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating, and Contacts:

Report presented in the absence of the chairman by C. K. Strobel, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for High-Resistivity, Low-Temperature-Coefficient Wire (B 267 – 53 T)

Adopted as Standard:

Rec. Practice for Dimensional Standards for Projection Welding Contacts (B 321 – 57 T)

Adopted as Standard, Revisions in:

Spec. for Drawn or Rolled Nickel-Chromium and Nickel-Chromium-Iron Alloys for Electrical Heating Elements (B 344 – 59)

Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes:

Report presented by C. H. Sample, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Preparation of Nickel for Electroplating with Nickel (B 343 – 60 T) (The vote in Committee B-8 on this recommendation was not available at the time of the session, and it was left that the vote should be reviewed by the Administrative Committee on Standards to determine whether there was a consensus.)

Adopted as Standard:

Rec. Practice for Preparation of Lead and Lead Alloys for Electroplating (B 319 – 57 T)
Rec. Practice for Preparation of Iron Castings for Electroplating (B 320 – 57 T)
Spec. for Electrodeposited Coatings of Lead on Steel (B 200 – 55 T)

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented in the absence of the chairman by G. L. Werley, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 58 T)

Adopted as Standard:

Spec. for Bronze Sintered Metal Powder Structural Parts (B 255 – 58 T)

Spec. for Brass Sintered Metal Powder Structural Parts (B 282 – 58 T)
Method for Subsieve Analysis of Granular Metal Powders by Air Classification (B 293 – 54 T)
Method for Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings (B 328 – 58 T)

Adopted as Standard, Revisions in:

Method of Sampling Finished Lots of Metal Powders (B 215 – 48)

Committee C-4 on Clay Pipe:

Report presented in the absence of the chairman by R. H. Welles, vice-chairman, and accepted as a report of progress.

Committee C-8 on Refractories:

Report presented in the absence of the chairman by L. C. Gilbert, ASTM staff, and the following actions taken subject to review by the Administrative Committee on Standards of the results of the vote in Committee C-8 since the vote was not available at the time of the session:

Accepted as Tentative:

Test for Hydration Resistance of Basic Brick (C 456 – 60 T)
Test for Disintegration of Carbon Refractories by Alkali (C 454 – 60 T)
Classification of Chrome Brick, Chrome-Magnesite Brick, Magnesite-Chrome Brick, and Magnesite Brick (C 455 – 60 T)

Accepted as Tentative, Revisions in:

Method of Testing Refractory Brick Under Load at High Temperatures (C 16 – 49)
Methods of Chemical Analysis of Refractory Materials (C 18 – 52)
Def. of Terms Relating to Refractories (C 71 – 55)
Spec. for Refractories for Incinerators (C 106 – 51)
Test for True Specific Gravity of Refractory Materials (C 135 – 47)

Adopted as Standard:

Methods for Chemical Analysis of Refractory Materials (C 18 – 56 T)
Test for Thermal Conductivity of Castable Refractories (C 417 – 58 T)
Classification of Fireclay and High-Alumina Refractory Brick (C 27 – 58 T)
Classification of Castable Refractories (C 401 – 57 T)
Classification of Silica Refractory Brick (C 416 – 58 T)

Withdrawal of Tentative Revision:

Methods of Chemical Analysis of Refractory Materials (C 18 – 52) (Tentative Revision issued June, 1956)

Committee C-17 on Asbestos-Cement Products:

Report presented by W. V. Friedlaender, chairman, and the following actions taken:

Accepted as Tentative:

Test for Organic Fiber Content of Asbestos-Cement Products (C 458 – 60 T)
Methods of Sampling and Testing Asbestos-Cement Flat Sheets, Roofing and Siding Shingles, and Clapboards (C 459 – 60 T)
Def. of Terms Relating to Asbestos-Cement and Related Products (C 460 – 60 T)

Adopted as Standard, Revisions in:

Spec. and Methods of Test for Flat Asbestos-Cement Sheets (C 220 – 55)
Spec. and Methods of Test for Asbestos-Cement Roofing Shingles (C 222 – 55)
Spec. and Methods of Test for Asbestos-Cement Siding Shingles and Clapboards (C 223 – 55)

The committee withdrew from the report as preprinted the proposed new tentative Methods of Testing Asbestos-Cement Pipe, and the proposed revisions of Tentative Specifications and Methods of Test for Asbestos-Cement Pressure Pipe (C 296 – 59 T) and Tentative Specifications and Methods of Test for Asbestos-Cement Non-Pressure Sewer Pipe (C 428 – 59 T).

Omitted from the report as preprinted was the recommendation that the tentative revisions of Sections 4(*b*) and 14(*c*), issued Sept. 9, 1958, of the Standard Specifications and Methods of Test for Asbestos-Cement Siding Shingles and Clapboards (C 223 – 55) be approved for reference to letter ballot of the Society for adoption as standard.

Committee C-24 on Joint Sealants:

Report presented by Wayne F. Koppes, chairman, and accepted as a report of progress.

Committee D-24 on Carbon Black:

Report presented by N. P. Bekema, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Carbon Black Used in Rubber Products (D 1765 – 60 T) (Jointly with Committee D-11)
Methods for Sampling Packaged Shipments of Carbon Black (D 1799 – 60 T)

Adopted as Standard:

Test for pH Value of Carbon Black (D 1512 - 57 T)
Test for Pour Density of Pelleted Carbon Black (D 1513 - 57 T)
Test for Sieve Residue from Carbon Black (D 1514 - 57 T)
Test for Sulfur Content of Carbon Black (D 1619 - 58 T)
Test for Fines Content of Pelleted Carbon Black (D 1508 - 57 T)
Test for Iodine Adsorption Number of Carbon Black (D 1510 - 57 T)
Test for Pellet Size Distribution of Carbon Black (D 1511 - 57 T)
Test for Volatile Content of Carbon Black (D 1620 - 58 T)

Tentative Withdrawn:

Test for Attrition of Pelleted Carbon Black (D 1507 - 57 T)

FIFTEENTH SESSION—GILLETT MEMORIAL LECTURE[11]

TUESDAY, JUNE 28, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT F. L. LAQUE

Ninth Gillett Memorial Lecture:

President LaQue opened the session by giving a brief statement of the purpose of the lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Institute to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first Director of the Institute, and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

President LaQue called upon H. C. Cross, Metallurgist and Assistant Technical Coordination Director, Battelle Memorial Institute, who introduced R. C. Dalzell, Assistant to the Director of the Division of Reactor Development, United States Atomic Energy Commission. Mr. Dalzell presented the ninth H. W. Gillett Memorial Lecture on the subject "Nuclear Fuel Element Development." Probably one of the most difficult problems facing the designers of nuclear reactors is that of standardization of fuel elements. Mr. Dalzell outlined the scientific problems and the engineering and economic considerations that are important in this developing industry. Research and development methods and observations were also discussed. He summarized the implication of all factors, including opportunities and prospects for standardization.

President LaQue expressed appreciation to Mr. Dalzell for his outstanding lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

---

[11] Issued as a separate publication.

SIXTEENTH SESSION—SOILS FOR ENGINEERING PURPOSES

TUESDAY, JUNE 28, 8:00 P.M.

SESSION CHAIRMAN: PHILIP KEENE

C. A. Hogentogler Award:

The Fifth C. A. Hogentogler Award was made to Robert L. Schiffman, Assistant Professor of Soil Mechanics, Rensselaer Polytechnic Institute, Troy, N. Y., and Charles R. Wilson, Engineer, Goodkind and O'Dea, Bloomfield, N. J., for their paper "The Mechanical Behavior of Chemically Treated Granular Soils," published in the 1958 ASTM *Proceedings*.

Papers:

Observed Settlement of Soft, Fine Grained Soils—E. J. Zegarra, presented by the author.

The Compressive Strength of Remolded Niagara Clay—D. J. Bazett and S. W. Smotrych, presented by Mr. Smotrych.

Duplication of Prototype Stress-Strain Relations by Laboratory Tests—R. G. Ahlvin and D. N. Brown, presented by Mr. Ahlvin.

The Importance of Freezing Rate on Frost Action in Soils—E. Penner, presented by Mr. R. F. Legget.

SEVENTEENTH SESSION—FATIGUE

TUESDAY, JUNE 28, 8:00 P.M.

SESSION CHAIRMAN: G. R. GOHN

Elevated Temperature Tensile and Fatigue Behavior of Unalloyed Arc-Cast Molybdenum—G. W. Brock and G. M. Sinclair, presented by Mr. Brock.

Fatigue Properties of Some High Strength Steels—H. E. Frankel, J. A. Bennett, and C. M. Carman, presented by Mr. Frankel.

Design Applications for Improving Fatigue Fatigue Resistance of Airplane Structures—C. R. Smith, presented by the author.

The Effect of Frequency and Physical Structure Upon the Solid Damping Property of Materials—H. L. Milligan, presented by title only.

(*Continued in Nineteenth Session*)

EIGHTEENTH SESSION—SYMPOSIUM ON RADIATION EFFECTS AND RADIATION DOSIMETRY[12]

WEDNESDAY, JUNE 29, 9:00 A.M.

SESSION CHAIRMAN: C. G. COLLINS

Determinant Sample Parameters in the Radiation Effects Testing of High Polymers—E. G. Fritz, presented by the author.

Irradiation Testing of Enrico Fermi Prototype Fuel Pins in the CP-5 Reactor—M. A. Silliman, A. A. Shoudy, P. R. Huebotter, and W. G. Blessing, presented by Mr. Shoudy.

The Effect of Neutron Irradiation on the Charpy-V and Drop-Weight Test Transition Temperatures of Various Steels and Weld Metals—J. R. Hawthorne and L. E. Steele, presented by Mr. Hawthorne.

Reactor Spectra Considerations in Radiation Effects Predictions—John Romanko, presented by the author.

Use of Radiochemistry and Mass Spectrometry Techniques for Fuel Depletion Studies—J. E. Hudgens, Jr., presented by the author.

(*Continued in Twenty-second Session*)

NINETEENTH SESSION—FATIGUE

(*Continued from the Seventeenth Session*)

WEDNESDAY, JUNE 29, 9:30 A.M.

SESSION CHAIRMAN: H. J. GROVER

New Observations Relating to the Mechanism of Fatigue Failure—M. A. Wilkov, presented by the author.

The R. R. Moore Rotating-Beam Fatigue Test—G. R. Gohn and A. Fox, presented by Mr. Gohn.

---

[12] Issued as *ASTM STP No. 286.*

Additional Studies of Effects of Anodic Coatings on Fatigue Strength of Aluminum Alloys—G. W. Stickley, presented by the author.

Relation Between Rolling-Contact Fatigue Life and Mechanical Properties Including Hardness for Several Aircraft Bearing Steels—E. V. Zaretsky and W. J. Anderson, presented by Mr. Anderson.

TWENTIETH SESSION—METALS

WEDNESDAY, JUNE 29, 9:30 A.M.

SESSION CHAIRMAN: VICE-PRESIDENT A. A. BATES

Long Time Atmospheric Corrosion Tests on Low Alloy Steels—H. R. Copson, presented by the author.

Effects of Alloying Additions on Hot Cracking of Austenitic Cr-Ni Stainless Steels—F. C. Hull, presented by the author.

Structural Beryllium—B. B. Muvdi, presented by the author.

Mechanical and Physical Properties of Five Copper-Base Casting Alloys—W. H. Johnson and J. G. Kura, presented by Mr. Johnson.

TWENTY-FIRST SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 29, 11:15 A.M.

SESSION CHAIRMAN: M. A. PINNEY

Committee D-7 on Wood:

Report presented by L. J. Markwardt, chairman, and the following actions taken:

Accepted as Tentative:

Methods of Testing Metal Fasteners (D 1761 - 60 T)

Method of Conducting Shear-Block Test for Quality Control of Glue Bonds in Scarf Joints (D 1759 - 60 T)

Method of Evaluating Wood Preservatives by Field Tests with Stakes (D 1758 - 60 T)

Method for Chemical Analysis of Wood Charcoal (D 1762 - 60 T)

Spec. for Pressure Treatment of Timber Products (D 1760 - 60 T)

Accepted as Tentative, Revisions in:

Test for Evaluating the Properties of Building Fiberboards (D 1037 - 56 T)

Def. of Terms Relating to Wood-Base Fiber and Particle Panel Materials (D 1554 - 58 T)

Adopted as Standard:

Test for Alpha-Cellulose in Wood (D 1103 - 55 T)

Test for Methoxyl Groups in Wood and Related Materials (D 1166 - 55 T)

Spec. for Modified Wood (D 1324 - 57 T)

Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses:

Report presented by H. R. Snoke, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 41)

Accepted as Tentative:

Test for Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (D 452 - 40) (Standard reverted to tentative without revision)

Adopted as Standard, Revisions in:

Spec. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (D 173 - 44)

Spec. for Asphalt Shingles Surfaced with Mineral Granules (D 225 - 58)

Spec. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-up Roofs (D 226 - 56)

Spec. for Asphalt Roll Roofing Surfaced with Mineral Granules (D 249 - 56)

Spec. for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-up Roofs (D 250 - 56)

Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 41)

The committee withdrew from the preprinted report the recommendation for adoption as standard without revision of the Specifications for Asphalt Insulating Siding Surfaced with Mineral Granules (D 1226 - 58 T) since this recommendation had appeared in the previous report of the committee.

Committee D-10 on Shipping Containers:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and accepted as a report of progress.

Committee D-12 on Soaps and Other Detergents:

Report presented in the absence of the chairman by R. J. Painter, ASTM staff, and the following actions taken:

Accepted for Publication as Information Only:

Method for Measuring Redeposition of Soil on Cotton Fabric.

Accepted as Tentative:

Test for Ethylene Diamine Tetraacetate (EDTA) in Soap or Synthetic Detergents (D 1767 - 60 T)
Test for Sodium Alkylbenzene Sulfonate in Synthetic Detergents by Ultraviolet Absorption (D 1768 - 60 T)

Accepted as Tentative, Revisions in:

Methods for Sampling and Chemical Analysis of Alkylbenzene Sulfonates (D 1568 - 58 T)
Methods for Sampling and Chemical Analysis of Fatty Alkyl Sulfates (D 1570 - 58T)
Spec. for Liquid Toilet Soap (D 799 - 51) (Standard reverted to tentative)

Adopted as Standard:

Spec. for Trisodium Phosphate (D 538 - 57 T)
Test for Copper in Soaps and Soap Products (D 460 - 57 T)

Committee D-13 on Textile Materials:

Report presented by B. L. Whittier, chairman, and the following actions taken:

Accepted as Tentative:

Test for Neps in Wool Top (D 1770 - 60 T)
Method of Sample Reweighing of Lots of Packaged Raw Wool (D 1771 - 60 T)
Method of Conditioning Textiles and Textile Products for Testing (D 1776 - 60 T)
Test for Imperfection Count of Textile Yarns (D 1772 - 60 T)
Method of Testing Elastic Fabrics (D 1775 - 60 T)
Method for Measuring Thickness of Textile Materials (D 1777 - 60 T)
Test for Dimensional Change of Knitted Fabrics in Laundering and Dimensional Restorability of the Laundered Fabric (D 1773 - 60 T)
Test for Elastic Properties of Uncrimped Fibers (D 1774 - 60 T)
Test for Colorfastness to Commercial Laundering and to Domestic Washing of Tufted Rugs and Carpets (D 1778 - 60 T)

Accepted as Tentative, Revisions in:

Methods for Identification of Fibers in Textiles (D 276 - 59 T)
Test for Vegetable Matter and Alkali-Insoluble Impurities in Scoured Wool (D 1113 - 54 T)
Test for Unevenness of Textile Strands (D 1425 - 59 T)
Test for Recovery of Textile Fabrics from Creasing, Using the Vertical Strip Apparatus (D 1295 - 53 T)
Test for Tuft Bind Pile Floor Coverings (D 1335 - 54 T)
Test for Linear Density of Textile Fibers by the Vibroscope (D 1577 - 58 T)
General Methods of Testing Woven Fabrics (D 39 - 59)
Def. of Terms Relating to Textile Materials (D 123 - 59)
Spec. and Methods of Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 - 40) (Standard reverted to tentative)
Test for Small Amounts of Copper and Manganese in Textiles (D 377 - 54) (Standard reverted to tentative)
Test for Strength of Cotton Fibers (Flat Bundle Method) (D 1445 - 57) (Standard reverted to tentative)

Adopted as Standard:

Test for Linear Density of Cotton Fibers (Array Sample) (D 1769 - 60)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Textile Materials (D 123 - 59)
Spec. and Methods of Test for Asbestos Yarns (D 299 - 54)
Test for Fiber Weight per Unit Length and Maturity of Cotton Fibers (Array Method) (D 1442 - 54)

Tentatives Withdrawn:

Methods of Testing Elastic Fabrics (D 1333 - 54 T)
Methods of Testing Wide Elastic Fabrics (D 1377 - 55 T)

The committee withdrew from the preprinted report the recommendations for adoption as standard without revision of the Tentative Methods of Testing Sewing Threads (D 204 - 57 T), and Tentative Method of Test for Abrasion Resistance of Textile Yarns (D 1379 - 55 T).

Committee D-14 on Adhesives

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Method for Climbing Drum Peel Test for Adhesives (D 1781 – 60 T)
Spec. for Adhesive for Acoustical Materials (D 1779 – 60 T)
Rec. Practice for Conducting Creep Tests of Metal-to-Metal Adhesives (D 1780 – 60 T)

Adopted as Standard:

Test for Consistency of Adhesives (D 1084 – 55 T)
Test for Adhesives Relative to Their Use as Electrical Insulation (D 1304 – 54 T)
Test for Amylaceous Matter in Adhesives (D 1488 – 57 T)
Test for Nonvolatile Content of Aqueous Adhesives (D 1489 – 57 T)
Test for Filler Content of Phenol, Resorcinol, and Melamine Adhesives (D 1579 – 58 T)
Test for Bonding Permanency of Water or Solvent Soluble Liquid Adhesives for Labeling Glass Bottles (D 1581 – 58 T)
Test for Nonvolatile Content of Phenol, Resorcinol and Melamine Adhesives (D 1582 – 58 T)
Test for Water Absorptiveness of Paper Labels (D 1584 – 58 T)
Spec. for Adhesives for Automatic Machine Labeling of Glass Bottles (D 1580 – 58 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Adhesives (D 907 – 55)

Committee D-15 on Engine Antifreezes:

Report presented by R. E. Vogel, chairman, and accepted as a report of progress.

Committee D-21 on Wax Polishes and Related Material:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Test for Accelerated Aging of Liquid Water-Emulsion Floor Polishes (D 1791 – 60 T)
Test for Water Spotting of Emulsion Floor Polishes (D 1793 – 60 T)
Test for Potential Long-Term Removability Properties of Water-Emulsion Floor Polishes (D 1792 – 60 T)

Committee D-22 on Methods of Atmospheric Sampling and Analysis:

Report presented in the absence of the chairman by F. Y. Speight, ASTM staff, and the following actions taken:

Accepted as Tentative:

Method for Collection and Analysis of Dustfall (D 1739 – 60 T)

Adopted as Standard:

Test for Concentration of Odorous Vapors (Adsorption Method) (D 1354 – 55 T)
Test for Inorganic Fluoride in the Atmosphere (D 1606 – 58 T)
Test for Nitrogen Dioxide and Nitric Oxide Content of the Atmosphere (Modified Griess-Ilosvay Reaction) (D 1607 – 58 T)
Test for Oxides of Nitrogen in Gaseous Combustion Products (Phenol-Disulfonic Acid Procedure) (D 1608 – 58 T)
Test for Oxidant (Ozone) Content of the Atmosphere (D 1609 – 58 T)
Methods for Continuous Analysis and Automatic Recording of the Sulfur Dioxide Content of the Atmosphere (D [illegible]355 – 58 T)
Def. of Terms Relating to Atmospheric Sampling and Analysis (D 1356 – 58 T)
Rec. Practices for Sampling Atmospheres for Analysis of Gases and Vapors (D 1605 – 58 T)

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and accepted as a report of progress.

TWENTY-SECOND SESSION—SYMPOSIUM ON RADIATION EFFECTS AND RADIATION DOSIMETRY[12]

(Continued from the Eighteenth Session)

WEDNESDAY, JUNE 29, 2:00 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT M. N. CLAIR

The Effect of Sample Parameters on Energy Absorption from a Neutron Beam—R. L. Johnston, presented by the author.

High Intensity Gamma Ray Dosimetry—D. Hale, D. R. Johnson, S. M. Dec, J. R. Coss, W. L. R. Rice, O. Van Sessoms, P. B. Hemmig, and R. E. Brocklehurst, presented by Mr. Van Sessoms.

A Solid State Bragg-Gray Cavity Chamber—V. H. Ritz and F. H. Attix, presented by Mr. Ritz.

Measurements of the Energy Absorbed from Pile Neutrons—D. Binder, C. D. Bopp, and R. L. Towns, presented by Mr. Binder.

Neutron Dosimetry for Materials Irradiation Studies—L. E. Steele and J. R. Hawthorne, presented by Mr. Steele.

Neutron Spectrum Determinations at Low Flux Levels—Paul Kruger, presented by the author.

TWENTY-THIRD SESSION—METALS

WEDNESDAY, JUNE 29, 2:00 P.M.

SESSION CHAIRMAN: C. L. KENT

Effect of Carbon Content and Melting Practice on the Room Temperature Sharp Edge Notch Tensile Characteristics of H-11 Modified and 300M Sheet Steels—J. L. Shannon, G. B. Espey, A. J. Repko, and W. F. Brown, Jr., presented by Mr. Brown.

Carbon:Strength Relationships in 5 per cent Chromium Ultra High Strength Steels—J. C. Hamaker and E. J. Vater, presented by Mr. Hamaker.

Spin-Fracture Tests on Ni-Mo-V Rotor Steels in the Brittle Fracture Range—G. O. Sankey, presented by the author.

Temperature Dependence of the Elastic Moduli of Several Stainless Steels—Frank Garofalo, presented by the author.

PANEL DISCUSSION ON LOW-CYCLE FATIGUE

WEDNESDAY, JUNE 29, 3:00 P.M.

This informal session was sponsored by the Applied Research Panel of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals.

TWENTY-FOURTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 29, 4:00 P.M.

SESSION CHAIRMAN: I. V. WILLIAMS

Committee A-1 on Steel:

Report presented by J. J. Kanter, chairman, and the following actions taken:

Accepted as Tentative:

Test for Notch Toughness of Turbine and Generator Steel Forgings (A 443 - 60 T)

Spec. for High-Strength Low Alloy Structural Manganese Vanadium Steel (A 441 - 60 T)

Spec. for Carbon Steel Plates with Improved Transition Properties (A 442 - 60 T)

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 - 57 T)

Spec. for General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy Steel Bars (A 29 - 59 T)

Spec. for Welded and Seamless Steel Pipe (A 53 - 59 T)

Spec. for Seamless Steel Boiler Tubes (A 83 - 59 T)

Spec. for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 58 T)

Spec. for Electric-Resistance-Welded Steel Pipe (A 135 - 59 T)

Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 58 T)

Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 59 T)

Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 - 58 T)

Spec. for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 - 58 T)

Spec. for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 59 T)

Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 58 T)

Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 58 T)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 58 T)

Spec. for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 58 T)

Spec. for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 59 T)

Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 59 T)

Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 58 T)

Spec. for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger and Condenser Tubes (A 249 – 59 T)

Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 58 T)

Spec. for Carbon Steel Bars Subject to Mechanical Property Requirements (A 306 – 57 T)

Spec. for Stress Relief Annealed Cold-Drawn Carbon Steel Bars (A 311 – 57 T)

Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 59 T) (Jointly with Committee A–10)

Spec. for Quenched and Tempered Carbon Steel Bars (A 321 – 59 T)

Spec. for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 59 T)

Spec. for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 – 59 T)

Spec. for Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 – 59 T)

Spec. for Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 59 T)

Spec. for Heavy-Walled Carbon and Low Alloy Steel Castings for Steam Turbines (A 356 – 58 T)

Spec. for Electric-Fusion-Welded Austenitic Chromium-Nickel Alloy Steel Pipe for High-Temperature Service (A 358 – 56 T)

Spec. for Ferritic Alloy Steel Forged and Bored Pipe for High-Temperature Service (A 369 – 55 T)

Spec. for Seamless Austenitic Steel Pipe for High-Temperature Central Station Service (A 376 – 59 T)

Spec. for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 59 T)

Spec. for Welded Large Outside Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service (A 409 – 58 T)

Spec. for Seamless and Electric Welded Low Alloy Steel Tubes for Economizers (A 423 – 58 T)

Spec. for Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 – 59 T)

Spec. for Quenched and Tempered Alloy Steel Bars, Hot-Rolled or Cold-Finished (A 434 – 59 T)

Spec. for Heat-Treated Carbon and Alloy Steel Track Bolts and Carbon Steel Nuts (A 183 – 55) (Standard reverted to tentative)

Spec. for Carbon Steel Seamless Drum Forgings (A 266 – 57) (Standard reverted to tentative)

Spec. for Carbon and Alloy Steel Forgings for Rings for Reduction Gears (A 290 – 55) (Standard reverted to tentative)

Spec. for Carbon and Alloy Steel Forgings for Pinions for Reduction Gears (A 291 – 55) (Standard reverted to tentative)

Adopted as Standard, Revisions in:

Spec. for Open-Hearth Steel Girder Rails of Plain, Grooved, and Guard Types (A 2 – 54)

Spec. for Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 27 – 58)

Spec. for Multiple-Wear Wrought Steel Wheels (A 57 – 57)

Spec. for Soft Steel Track Spikes (A 65 – 54)

Spec. for Austenitic Manganese-Steel Castings (A 128 – 33)

Spec. for High-Strength Steel Castings for Structural Purposes (A 148 – 58)

Spec. for One-Wear Wrought Steel Wheels (A 186 – 58)

Spec. for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 56)

Spec. for High Strength Low Alloy Structural Steel (A 242 – 55)

Spec. for Welded and Seamless Open-Hearth Iron Pipe (A 253 – 58)

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 58) (Jointly with Committee A–10)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 59) (Jointly with Committee A-10)

Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 58) (Jointly with Committee A-10)

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 59) (Jointly with Committee A-10)

Spec. for Carbon and Alloy Steel Forgings for Pressure Vessel Shells (A 372 – 58)

The committee withdrew from the preprinted report the revision of Tentative Specifications for Galvanized Steel Transmission Tower Bolts (A 394 – 55 T), and the revision for immediate adoption of Standard Specification for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 – 59).

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by Bruce W. Gonser, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Remelted Lithium Metal in Ingot Form (B 357 – 60 T)

Accepted as Tentative, Revisions in:

Spec. for Solder Metal (B 32 – 58 T)
Spec. for Rosin Flux Cored Solder (B 284 – 58 T)

Adopted as Standard:

Spec. for Fire-Refined Casting Copper (B 72 – 55 T)

The committee withdrew from the preprinted report the proposed tentative Specification for Primary Columbium Metal.

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by W. H. Jennings, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Copper-Nickel Alloys in Ingot, Slab, or Shot Form (B 358 – 60 T)

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Wrought Copper-Alloy Wire (B 250 – 58a T)
Spec. for Flat Copper Products with Finished (Rolled or Drawn) Edges (Flat Wire and Strip) (B 272 – 58 T)

Adopted as Standard:

Spec. for Copper and Copper-Alloy Seamless Condenser and Heat Exchanger Tubes with Integral Fins (B 359 – 60)
Spec. for Hard-Drawn Copper Capillary Tube for Restriction Applications (B 360 – 60)
Spec. for Copper-Zinc-Manganese Alloy (Manganese Brass) Sheet and Strip (B 291 – 58 T)

Adopted as Standard, Revisions in:

Spec. for Free-Cutting Brass Rod, Bar, and Shapes for Use in Screw Machines (B 16 – 58)
Spec. for Brass Plate, Sheet, Strip, and Rolled Bar (B 36 – 56)
Spec. for Steam or Valve Bronze Castings (B 61 – 52)
Spec. for Composition Brass or Ounce Metal Castings (B 62 – 52)
Spec. for Seamless Copper Tube, Bright Annealed (B 68 – 58)
Spec. for Seamless Copper Tube (B 75 – 59)
Spec. for Phosphor Bronze Plate, Sheet, Strip, and Rolled Bar (B 103 – 55)
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 58)
Spec. for Leaded Brass Plate, Sheet, Strip, and Rolled Bar (B 121 – 55)
Spec. for Copper-Nickel-Zinc Alloy (Nickel Silver) and Copper-Nickel Alloy Plate, Sheet, Strip, and Rolled Bar (B 122 – 59)
Spec. for Copper Rod, Bar, and Shapes (B 133 – 58)
Spec. for Seamless Brass Tube (B 135 – 58)
Spec. for Copper Sheet, Strip, Plate, and Rolled Bar (B 152 – 58)
Spec. for Copper Bus Bar, Rod, and Shapes (B 187 – 58)
Spec. for Seamless Copper Bus Pipe and Tube (B 188 – 59)
Spec. for General Requirements for Wrought Copper and Copper-Alloy Bar, Rod, and Shapes (B 249 – 59)
Spec. for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 59)
Spec. for Seamless Copper Tube for Refrigeration Field Service (B 280 – 59)
Spec. for Threadless Copper Pipe (B 302 – 59)

The committee withdrew from the preprinted report the revisions for immediate adoption of Standard Specifications for Tin Bronze and Leaded Tin Bronze Sand Castings (B 143 – 52), and Standard Specifications for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 52).

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman, and the following action taken:

Adopted as Standard:

Spec. for Aluminum-Alloy Die Castings (B 85 – 58 T)

Committee E-3 on Chemical Analysis of Metals:

Report presented by Arba Thomas, chairman, and the following actions taken:

Accepted as Tentative:

Methods for Chemical Analysis of Ferroboron (E 31 – 60 T)
Methods for Chemical Analysis of Electronic Nickel (E 107 – 60 T)
Test for Hydrogen Loss of Copper Powder (E 159 – 60 T)

Accepted as Tentative, Revisions in:

Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 59 T)
Methods for Chemical Analysis of Titanium and Titanium-Base Alloys (E 120 – 56 T)
Rec. Practice for Photometric Methods for Chemical Analysis of Metals (E 60 – 50 T)

Adopted as Standard:

Methods for Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 35 – 58 T)
Methods for Chemical Analysis of White Metal Bearing Alloys (E 57 – 57 T)
Methods for Chemical Analysis of Electronic Nickel (E 107 – 56 T)
Methods for Chemical Analysis of Copper-Chromium Alloys (E 118 – 56 T)
Methods for Chemical Analysis of Copper-Tellurium Alloys (E 121 – 56 T)
Method for Determination of Arsenic in Fire-Refined Copper (E 62 – 58 T)
Methods for Chemical Analysis of Titanium and Titanium-Base Alloys (E 120 – 56 T)

Adopted as Standard, Revisions in:

Rec. Practices for Apparatus and Reagents for Chemical Analysis of Metals (E 50 – 53)

Committee E-13 on Absorption Spectroscopy:

Report presented in the absence of the chairman by R. F. Robey, secretary, and the following actions taken:

Accepted as Tentative:

Rec. Practice for General Techniques of Infrared Quantitative Analysis (E 168 – 60 T)
Rec. Practice for General Techniques of Ultraviolet Quantitative Analysis (E 169 – 60 T)

TWENTY-FIFTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 29, 4:00 P.M.

SESSION CHAIRMAN: W. R. WILLETS

Committee C-7 on Lime:

Report presented in the absence of the acting chairman by H. F. Kriege, and the following action taken:

Accepted as Tentative, Revisions in:

Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 – 58)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by W. H. Price, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete (C 457 – 60 T)

Accepted as Tentative, Revisions in:

Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (C 116 – 59 T)
Test for Volume Change of Cement Mortar and Concrete (C 157 – 54 T)
Spec. for Waterproof Paper for Curing Concrete (C 171 – 59 T)
Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 – 58 T)
Method of Testing Air-Entraining Admixtures for Concrete (C 233 – 58 T)
Spec. for Air-Entraining Admixtures for Concrete (C 260 – 58 T)
Spec. for Lightweight Aggregates for Structural Concrete (C 330 – 59 T)
Spec. for Fly Ash for Use as an Admixture in Portland Cement Concrete (C 350 – 57 T)

Adopted as Standard:

Test for Unit Weight of Aggregate (C 29 – 55 T)
Test for Organic Impurities in Sands for Concrete (C 40 – 56 T)
Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 – 56 T)
Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215 – 58 T)
Spec. for Packaged, Dry, Combined Materials for Mortar and Concrete (C 387 – 58 T)

Adopted as Standard, Revisions in:

Methods of Securing, Preparing, and Testing

Specimens from Hardened Concrete for Compressive and Flexural Strengths (C 42 - 59)

Committee C-21 on Ceramic Whitewares and Related Products:

Report presented in the absence of the chairman by L. C. Gilbert, ASTM staff, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Ceramic Whitewares and Related Products (C 242 - 58 T)

Adopted as Standard:

Test for Crazing Resistance of Fired Glazed Whitewares by Autoclave Treatment (C 424 - 58 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Ceramic Whitewares and Related Products (C 242 - 56)

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Reported presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Isobutyl Alcohol (Isobutanol) (D 1719 - 60 T)
Specs. for Isobutyl Acetate (93 Per cent Minimum Ester Grade) (D 1718 - 60 T)
Method for Qualitative Determination of Fish Oil in Drying Oils and Drying Oil Fatty Acids (D 1724 - 60 T)
Test for Water Miscibility of Isopropyl Alcohol (Isopropanol) (D 1722 - 60 T)
Test for Water Solubility of Acetone (D 1723 - 60 T)
Test for Permanganate Time of Tricresyl Phosphate (D 1721 - 60 T)
Test for Dilution Ratio in Cellulose Nitrate Solutions for Active Solvents, Hydrocarbon Diluents, and Cellulose Nitrates (D 1720 - 60 T)
Test for Hiding Power of Nonchromatic Paints (D 1738 - 60 T)
Test for Phthalate Ester Color of High-Gravity Glycerine (D 1728 - 60 T)
Test for Viscosity of Resin Solutions (D 1725 - 60 T)
Test for Urea Content of Nitrogen Resins (D 1727 - 60 T)
Test for Hydrolyzable Chlorine Content of Liquid Epoxy Resins (D 1726 - 60 T)
Test for Efflorescence of Interior Latex Paints (D 1736 - 60 T)
Test for Elongation of Attached Coatings of Paint, Varnish, Lacquer, and Related Products with Cylindrical Mandrel Apparatus (D 1737 - 60 T)
Method for Preparation of Aluminum Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 1733 - 60 T)
Method of Making and Preparing Concrete and Masonry Panels for Testing Paint Finishes (D 1734 - 60 T)
Method for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting (D 1730 - 60 T)
Method for Preparation of Magnesium Alloy Surfaces for Painting (D 1732 - 60 T)
Method for Water Fog Testing of Organic Coatings (D 1735 - 60 T)
Methods for Preparation of Hot-Dip Aluminum Surfaces for Painting (D 1731 - 60 T)
Rec. Practice for Visual Evaluation of Color Differences of Opaque Materials (D 1729 - 60 T)

Accepted as Tentative, Revisions in:

Test for Color Difference Using the Hunter Color Difference Meter (D 1365 - 55 T)
Test for Color Difference Using the Color Eye (D 1495 - 57 T)
Test for Color Change of White Architectural Enamels (D 1543 - 58 T)
Test for Aniline Point and Mixed Aniline Point of Hydrocarbon Solvents (D 1012 - 51) (Standard reverted to tentative)

Adopted as Standard:

Specs. for Distilled Soybean Fatty Acids (D 1537 - 58 T)
Spec. for Distilled Linseed Fatty Acids (D 1538 - 58 T)
Spec. for Dehydrated Castor Acids (D 1539 - 58 T)
Test for Mass Color and Tinting Strength of Color Pigments (D 387 - 52 T)
Test for Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (D 1211 - 57 T)
Test for Unsaponifiable Matter in Alkyd Resins and Resin Solutions (D 1397 - 58 T)
Test for Volatile Matter in Tricresyl Phosphate (D 1468 - 57 T)
Test for Density of Paint, Varnish, Lacquer, and Related Products (D 1475 - 57 T)
Test for Heptane Miscibility of Lacquer Solvents (D 1476 - 57 T)
Test for Total Iodine Value of Drying Oils and Their Derivatives (D 1541 - 58 T)
Test for Viscosity of Transparent Liquids by Bubble Time Method (D 1545 - 58 T)
Test for Melamine Content of Nitrogen Resins (D 1597 - 58 T)

Test for Acetone in Methyl Alcohol (Methanol) (D 1612 – 58 T)
Test for Alkalinity in Acetone (D 1614 – 58 T)
Test for Glycerol, Ethylene Glycol, and Pentaerythritol in Alkyd Resins (D 1615 – 58 T)
Test for Copper Corrosion by Mineral Spirits (Copper Strip Test) (D 1616 – 58 T)
Test for Gas Checking and Draft Test of Varnish Films (D 1643 – 59 T)
Rec. Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 – 57 T)
Spec. for Refined Soybean Oil (D 1462 – 57 T)
Test for Oil Absorption of Pigments by Gardner-Coleman Method (D 1483 – 57 T)
Method for Qualitative Tests for Rosin in Varnishes (D 1542 – 58 T)
Test for Ester Value of Lacquer Solvents and Diluents (D 1617 – 58 T)

Adopted as Standard, Revisions in:

Spec. for Methyl Ethyl Ketone (D 740 – 56)
Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 40)
Test for Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus (D 522 – 41)

Committee D-9 on Electrical Insulating Materials:

Report presented by H. K. Graves, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation (D 202 – 55 T)
Methods of Testing Nonrigid Vinyl Chloride Polymer Tubing (D 876 – 59 T)
Methods of Testing Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (D 1000 – 59 T)
Methods for Sampling and Testing Untreated Mica Paper Used for Electrical Insulation (D 1677 – 59 T)

Tentative Withdrawn:

Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 54 T)

The committee withdrew from the preprinted report the recommendation for revision and reversion to tentative of the Standard Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 – 51) since this method is now under the sole jurisdiction of Committee D-20 on Plastics.

Committee D-17 on Naval Stores:

Report presented by S. R. Snider, chairman, and accepted as a report of progress.

Committee D-18 on Soils for Engineering Purposes:

Report presented in the absence of the chairman by W. S. Housel, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Method for Grain-Size Analysis of Soils (D 422 – 59 T)

Adopted as Standard:

Def. of Terms and Symbols Relating to Soil Mechanics (D 653 – 58 T)

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

Report presented in the absence of the chairman by C. L. Guettel, secretary, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Testing Electron Tube Parts Using a Reference Planar Diode (F 11 – 60 T)

Adopted as Standard:

Test for Volatile Content of Germanium Oxide (F 5 – 58 T)
Test for Bulk Density of Germanium Dioxide (F 6 – 58 T)
Test for Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (F 205 – 58T)
Test for Sag of Tungsten Wire (F 269 – 58 T)
Spec. for Molybdenum Wire Under 20 Mils in Diameter (F 289 – 56 T)

Committee E-5 on Fire Tests of Materials and Construction:

The report, presented by W. J. Krefeld, chairman, contained the following recommendations:

For Publication as Information Only:

Method of Test for Surface Flammability of Building Materials Using an 8-ft Tunnel Furnace

New Tentatives:

Methods of Fire Tests of Window Assemblies
Test for Measuring the Surface Flammability of Materials Using a Radiant Heat Energy Source

Def. of Terms Relating to Fire Tests of Building Construction and Materials

Adoption of Tentative as Standard with Revisions:

Test for Surface Burning Characteristics of Building Materials (E 84 - 59 T)

Adoption of Tentative Revisions as Standard:

Methods of Fire Tests of Building Construction and Materials (E 119 - 58)

Objection was raised at the meeting to the adoption as standard of the revised Tentative Method E 84 - 59 T. During discussion of a motion to refer this recommendation back to Committee E-5 for further consideration, the question of quorum was raised. On the basis of a count of members present, the chairman of the session ruled the absence of a quorum and accordingly no actions were taken on the recommendations in the report.

TWENTY-SIXTH SESSION—MARBURG LECTURE,[13] MEDAL AWARDS

WEDNESDAY, JUNE 29, 4:30 P.M.

SESSION CHAIRMAN: PRESIDENT F. L. LAQUE

Charles B. Dudley Medal Award:

The Thirtieth Charles B. Dudley Medal Award was made to G. R. Irwin, J. A. Kies, and H. L. Smith, Mechanics Division Superintendent, Ballistics Branch Head and Fracture Section Head, respectively, U. S. Naval Research Laboratory, Washington, D. C., for their paper on "Fracture Strengths Relative to Onset and Arrest of Crack Propagation," published in the 1958 *Proceedings*.

Richard L. Templin Award:

The Fourteenth Richard L. Templin Award was made to J. W. Shupe, Associate Professor, Kansas State College, Manhattan, Kans., and W. H. Goetz, Research Engineer, Purdue University, Lafayette, Ind., for their paper on "A Laboratory Method for Determining the Skidding Resistance of Bituminous Paving Mixtures," published in the 1958 *Proceedings*.

Sam Tour Award:

The Ninth Sam Tour Award was made to P. J. Sereda, Building of Materials Section, National Research Council of Canada, Ottawa, Canada, for his paper on "Measurements of Surface Moisture," published in the ASTM BULLETIN, February, 1958.

Edgar Marburg Lecture:

A brief statement of the purpose of the lecture was given by President LaQue. Farrington Daniels, Professor Emeritus of the University of Wisconsin and Vice-President of the National Academy of Sciences, was then introduced by President LaQue as the thirty-fourth Edgar Marburg Lecturer. Speaking on the subject "Solar Energy," Mr. Daniels discussed the abundance and limitations of solar energy, together with a review of solar energy research, emphasizing particularly the new materials that are necessary in the successful application of solar energy. The research review included the subjects of heating, cooling, distillation of salt water, solar engines, photoelectricity, thermal electricity, and photochemistry. New materials that were discussed included: (1) plastics for reflectors and covers for solar collectors; (2) silicon in solar cells; (3) solid-state devices for thermoelectric and thermionic energy converters; (4) refrigerant systems for possible application in solar cooling cycles; and (5) selective coatings for attaining high efficiency energy absorbers.

President LaQue expressed appreciation to Mr. Daniels for his excellent lecture and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

---

[13] Issued as a separate publication.

ANNUAL DINNER

WEDNESDAY, JUNE 29, 6:30 P.M.

The Philadelphia District, through its Council, acted as the official host at the Cocktail Party and Dinner which was followed by entertainment. Approximately 360 persons attended this function.

PAPERS ON EFFECT OF NUCLEAR BOMBARDMENT ON RUBBER MATERIALS

THURSDAY, JUNE 30, 9:00 A.M.

This informal session was sponsored by Committee D-11 on Rubber and Rubber-Like Materials. The following papers were presented:

The Factor of Ozone Generation in Tests of Radiation Damage to Elastomers—L. B. Bang.

Good and Bad Effects of Nuclear Radiation on Rubber—J. W. Born.

TWENTY-SEVENTH SESSION—SYMPOSIUM ON LOW-TEMPERATURE PROPERTIES OF HIGH-STRENGTH AIRCRAFT AND MISSILE MATERIALS[14]

THURSDAY, JUNE 30, 9:30 A.M.

SESSION CHAIRMAN: J. F. WATSON

Sharp-Notch Behavior of Some High-Strength Sheet Aluminum Alloys and Welded Joints at 75, −320 and −423 F—M. P. Hanson, G. W. Stickley, and H. T. Richards, presented by Mr. Hanson.

Some Low-Temperature Properties of Aluminum Alloy Weldments—W. R. Lucas and C. E. Cataldo, presented by Mr. Cataldo.

Thermal Cycling Under Constant Load to Low Temperature of Aluminum and Magnesium Alloys—Volker Weiss, G. T. Schaeffer, A. Saule, and George Sachs, presented by Mr. Weiss.

Low-Temperature Properties of Cast and Wrought Magnesium Alloys—R. W. Fenn, Jr., presented by the author.

---

[14] To be issued as *ASTM STP No. 287.*

(Continued in Thirtieth and Thirty-Second Sessions)

TWENTY-EIGHTH SESSION—GENERAL TESTING

THURSDAY, JUNE 30, 9:30 A.M.

SESSION CHAIRMAN: R. D. THOMPSON

A New Impact Detonability Tester for Evaluating Materials with Highly Reactive Oxidizers—R. Kopituk, presented by the author.[15]

Shock Testing Method for Ground Support Equipment and Industrial Items Weighing From 1000 to 50,000 Pounds—W. H. Grumet, presented by the author.[16]

Flow Properties of Bulk Solids—A. W. Jenike, P. J. Elsey, and R. H. Woolley, presented by Mr. Jenike.

A Notched Ring Specimen for Hydrogen Embrittlement Studies—F. S. Williams, W. Beck, and E. J. Jankowsky, presented by Mr. Williams.

---

[15] Published in the ASTM BULLETIN No. 250, Dec., 1960.

[16] To be published in *Materials Research & Standards* (formerly ASTM BULLETIN).

TWENTY-NINTH SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 30, 11:15 A.M.

SESSION CHAIRMAN: E. C. SHUMAN

Committee C-3 on Chemical-Resistant Mortars:

Report presented in the absence of the chairman by W. C. Hansen and the following actions taken:

Accepted as Tentative:

Test for Flexural Strength of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 453 - 60 T)

Accepted as Tentative, Revisions in:

Rec. Practice for Use of Hydraulic Cement Mortars in Chemical-Resistant Masonry (C 398 - 57 T)

Adopted as Standard:

Rec. Practice for Installing Sulfur Mortar Joints (C 386 - 59 T)
Spec. for Resin-Type Chemical-Resistant Mortars (C 395 - 57 T)
Test for Compressive Strength of Chemically Setting, Silicate-Type Chemical-Resistant Mortars (C 396 - 57 T)
Rec. Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 - 57 T)
Rec. Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 - 59 T)

Adopted as Standard, Revisions in:

Test for Compressive Strength of Resin-Type Chemical-Resistant Mortars (C 306 - 55)

Committee C-13 on Concrete Pipe:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Reinforced-Concrete Culvert, Storm Drain, and Sewer Pipe (C 76 - 59 T)
Spec. for Joints for Circular Concrete Sewer and Culvert Pipe Using Flexible, Watertight, and Rubber-Type Gaskets (C 443 - 59 T)
Spec. for Perforated Concrete Pipe (C 444 - 59 T)

Adopted as Standard:

Spec. for Concrete Drain Tile (C 412 - 58 T)

Adopted as Standard, Revisions in:

Spec. for Concrete Pipe for Irrigation or Drainage (C 118 - 59)

Committee C-16 on Thermal Insulating Materials:

Report presented in the absence of the chairman by E. C. Shuman, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Thermal Insulating Materials (C 168 - 56)

Adopted as Standard:

Spec. for Structural Insulating Board Made From Vegetable Fibers (C 208 - 57 T)
Spec. for Mineral Wool Block or Board Thermal Insulation for Elevated Temperatures (C 392 - 57 T)
Test for Thermal Conductance and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (C 236 - 54 T)
Test for Linear Shrinkage of Preformed High-Temperature Thermal Insulation Subjected to Soaking Heat (C 356 - 55 T)
Test for Consistency of Wet-Mixed Thermal Insulating Cement (C 405 - 57 T)
Method of Sampling Preformed Thermal Insulation (C 390 - 57 T)
Method of Testing Structural Insulating Board Made from Vegetable Fibers (C 209 - 57 T)

Committee C-22 on Porcelain Enamel:

Report presented in the absence of the chairman by M. Bozsin, and the following actions taken:

Adopted as Standard:

Test for Fusion Flow of Porcelain Enamel Frits (Flow-Button Method) (C 374 - 55 T)
Test for Torsion Resistance of Laboratory Specimens of Porcelain-Enameled Iron and Steel (C 409 - 57 T)

Committee E-1 on Methods of Testing:

Report presented in the absence of the chairman by D. E. Parsons, vice-chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Precision Micromesh Sieves (E 161 - 60 T)

Accepted as Tentative, Revisions in:

Spec. for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (E 11 - 58 T)
Spec. for Apparatus for Determination of Water by Distillation (E 123 - 56 T)
Spec. for ASTM Thermometers (E 1 - 59)

Adopted as Standard:

Methods for Notched Bar Impact Testing of Metallic Materials (E 23 - 56 T)

Adopted as Standard, Revisions in:

Spec. for ASTM Thermometers (E 1 - 59)
Method of Determining Relative Humidity (D 337 - 34) (Redesignated E 157 - 60)

Committee E-6 on Methods of Testing Building Constructions:

Report presented in the absence of the chairman by E. C. Shuman, and the following action taken:

Accepted as Tentative, Revisions in:

Methods of Testing Materials for Use as Vapor Barriers Under Concrete Slabs and as Ground Cover in Crawl Spaces (E 154 - 59 T)

Committee E-14 on Mass Spectrometry:

Report presented by R. A. Friedel, chairman, and accepted as a report of progress.

THIRTIETH SESSION—SYMPOSIUM ON LOW-TEMPERATURE PROPERTIES OF HIGH-STRENGTH AIRCRAFT AND MISSILE MATERIALS[14]

(*Continued from the Twenty-Seventh Session*)

THURSDAY, JUNE 30, 2:30 P.M.

SESSION CHAIRMAN: C. L. CLARK

Some Mechanical Properties of Magnesium Alloys at Low Temperatures—R. P. Reed, R. P. Mikesell, and R. L. Greeson, presented by Mr. Reed.
Sharp-Edge-Notch Tensile Characteristics of Several High-Strength Titanium Sheet Alloys at Room and Cryogenic Temperatures—G. B. Espey, M. H. Jones, and W. F. Brown, Jr., presented by Mr. Brown.
Materials for Use at Liquid Hydrogen Temperatures—J. H. Belton, L. L. Godby, and B. L. Taft, presented by Mr. Taft.
Effect of Stress Concentration on the Tensile Strength of Titanium and Steel Sheet Alloys—George Sachs and J. G. Sessler, presented by Mr. Sessler.

(*Continued in Thirty-Second Session*)

THIRTY-FIRST SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 30, 4:30 P.M.

SESSION CHAIRMAN: R. W. SENIFF

Committee A-3 on Cast Iron:

Report presented by T. E. Eagan, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Austenitic Nodular Iron Castings (A 439 - 60 T)
Spec. for Ferritic Nodular Iron Castings for Valves, Flanges, Pipe Fittings, and Other Piping Components (A 445 - 60 T)

Adopted as Standard:

Specs. for Foundry Pig Iron (A 43 - 55 T)

Adopted as Standard, Revisions in:

Methods of Chill Testing of Cast Iron (A 367 - 58)

Committee A-5 on Corrosion of Iron and Steel:

Report presented by H. F. Hormann, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets for Culverts and Underdrains (A 444 -

60 T), with a number of editorial changes including rearrangement of some of the sections, as well as revisions of the headings as follows: Base Metal Manufacture was changed to Base Metal; Sampling was changed to Sampling by Sheet Purchaser; and Test Specimens was changed to Sampling by Sheet Producer.

Adopted as Standard, Revisions in:

Spec. for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 59)

Committee D-5 on Coal and Coke:

Report presented in the absence of the chairman by R. L. Coryell, secretary, and the following actions taken:

Accepted as Tentative:

Test for Sulfur in Coal Ash (D 1757 – 60 T)
Test for Carbon Dioxide in Coal (D 1756 – 60 T)

Adopted as Standard:

Test for Cubic Foot Weight of Crushed Bituminous Coal (D 291 – 58 T)

The committee withdrew from the preprinted report the proposed Tentative Method of Test for Plastic Properties of Coal by the Gieseler Plastometer.

Committee D-6 on Paper and Paper Products:

Report presented in the absence of the chairman by H. A. Birdsall, vice-chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Interlaboratory Evaluation of Test Methods Used with Paper and Paper Products (D 1749 – 60 T)

Adopted as Standard:

Test for Chloride Content of Paper and Paper Products (D 1161 – 51 T)

Adopted as Standard, Revisions in:

Test for Tensile Breaking Strength of Paper and Paper Products (D 828 – 48)
Test for Ring Crush of Paperboard (D 1164 – 53)

Committee D-23 on Cellulose and Cellulose Derivatives:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Test for Intrinsic Viscosity of Cellulose (D 1795 – 60 T)
Test for Cellulose Chain Length Uniformity by Fractional Precipitation of Cellulose Nitrates (D 1716 – 60 T)
Test for Alcohol-Benzene Soluble Matter in Cellulose (D 1794 – 60 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Cellulose and Cellulose Derivatives (D 1695 – 59 T)

Committee D-25 on Casein and Similar Protein Materials:

Report presented in the absence of the chairman by S. F. Etris, ASTM staff, and the following actions taken:

Accepted as Tentative:

Test for Ash in Casein and Isolated Soy Protein (D 1797 – 60 T)
Test for Fixed Ash in Casein and Isolated Soy Protein (D 1798 – 60 T)
Test for Oil in Casein and Isolated Soy Protein (D 1804 – 60 T)
Test for Free Acidity in Casein and Isolated Soy Protein (D 1801 – 60 T)
Test for Moisture in Casein and Isolated Soy Protein (D 1802 – 60 T)
Test for Nitrogen in Casein and Isolated Soy Protein (D 1803 – 60 T)
Test for Alkali Requirement of Casein and Isolated Soy Protein (D 1800 – 60 T)

Committee D-26 on Halogenated Organic Solvents:

Report presented by V. E. Amspacher, chairman, and accepted as a report of progress.

Committee E-10 on Radioisotopes and Radiation Effects:

Report presented by C. E. Webber, chairman, and the following action taken:

Accepted as Tentative:

Def. of Terms Relating to Dosimetry (E 170 – 60 T), with the deletion of the definition for "Fast Neutrons."

The committee withdrew from the preprinted report the proposed Tentative Method of Test for Sulfur in Hydrocarbons by Iron-55 X-ray Absorption, Method for Radiometric Determination of Magnesium Oxide in Cement, and Method for Measurement by Nuclear Means of Moisture and Density in Soils.

Committee E-12 on Appearance:

Report presented by G. W. Ingle, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Goniophotometry of Transmitting Objects and Materials (E 166 - 60 T)

Rec. Practice for Goniophotometry of Reflecting Objects and Materials (E 167 - 60 T)

THIRTY-SECOND SESSION—SYMPOSIUM ON LOW-TEMPERATURE PROPERTIES OF HIGH-STRENGTH AIRCRAFT AND MISSILE MATERIALS[14]

(*Continued from the Thirtieth Session*)

THURSDAY, JUNE 30, 8:00 P.M.

SESSION CHAIRMAN: T. N. ARMSTRONG

Mechanical Properties of High Strength 301 Stainless Steel Sheet at 70, −320, and −423 F in the Base Metal and Welded Joint Configuration—J. F. Watson and J. L. Christian, presented by title only.

Effect of Zerolling on the Properties of Modified Type 347—C. R. Mayne, presented by the author.

Properties of the Precipitation-Hardening Stainless Steels and Low-Alloy High-Strength Steels at Very Low Temperatures—J. E. Campbell and L. P. Rice, presented by Mr. Rice.

Low-Temperature Properties of Cold-Rolled AISI Types 301, 302, 304ELC and 310 Stainless Steel Sheet—J. F. Watson and J. L. Christian, presented by Mr. Watson.

The Effect of Carbon Content on the Notch Properties of 43XX-Vanadium Modified and 5 per cent Cr Sheet Steels—E. P. Klier, presented by the author.

Summary—A. Hurlich, presented by the author.

THIRTY-THIRD SESSION—ROAD AND PAVING MATERIALS[17]

THURSDAY, JUNE 30, 8:00 P.M.

SESSION CHAIRMAN: E. A. WHITEHURST

Stress Deformation Characteristics of Sand-Bituminous Mixtures—J. A. Carpenter, presented by the author.

The Effect of Compaction Temperature on the Properties of Bituminous Concrete—R. W. Kiefer, presented by the author.

A Method for Measuring Air Permeabilities of Asphalt Concrete Pavements—W. H. Ellis and R. J. Schmidt, presented by Mr. R. S. Winniford.

The Influence of Asphalt Composition on Its Rheology—R. S. Winniford, presented by the author.

Emulsified Asphalt Tests and Specifications—K. E. McConnaughay, presented by Mr. J. D. Spahr.

Laboratory Tests and Recommended Specifications for Rapid Setting and Coarse Aggregate Mixing Cationic Asphalt Emulsions—E. W. Mertens, L. D. Coyne, and E. C. Rogers, presented by Mr. Mertens.

---

[17] Issued as *ASTM STP No. 294*.

THIRTY-FOURTH SESSION—CEMENT AND PLASTER

THURSDAY, JUNE 30, 8:00 P.M.

SESSION CHAIRMAN: WILLIAM LERCH

The Reproducibility of Flexure and Compression Tests for Determining the Strength of Cement—M. F. Kaplan, presented by the author.

An X-ray Diffraction Investigation of Hydrated Portland Cement Pastes—D. L. Kantro, L. E. Copeland, and Elaine R. Anderson, presented by Mr. Kantro.

An Improved Method for the Determination of the Normal Consistency of Gypsum Plasters—R. A. Kuntze, presented by the author.[18]

Effect of High Temperatures on Concretes Incorporating Different Aggregates—N. G. Zoldners, presented by the author.

---

[18] Published in the ASTM BULLETIN, No. 246, May, 1960, p. 35.

THIRTY-FIFTH SESSION—SYMPOSIUM ON QUALITY OF OBSERVATIONS[19]

FRIDAY, JULY 1, 9:30 A.M.

SESSION CHAIRMAN: A. T. MCPHERSON

On the Meaning of Precision and Accuracy—R. B. Murphy, presented by the author.

How to Evaluate Precision—W. S. Connor, presented by the author.

Some Observations on Evaluating Accuracy—W. J. Youden, presented by the author.

On the Analysis of Planned Experiments—Milton Terry, presented by the author.

---

[19] Published in *Materials Research Standards* (formerly ASTM BULLETIN).

THIRTY-SIXTH SESSION—COMMITTEE REPORT SESSION

FRIDAY, JULY 1, 12:30 P.M.

SESSION CHAIRMAN: H. M. HANCOCK

Committee D-2 on Petroleum Products and Lubricants:

Report presented by H. M. Smith, chairman, and the following actions taken:

Accepted as Tentative:

Test for Oil Separation from Lubricating Grease During Storage (D 1742 - 60 T)

Test for Rust Preventive Properties of Lubricating Greases (D 1743 - 60 T)

Test for Functional Life of Ball Bearing Greases (D 1741 - 60 T) with the addition of the following new Section 2 on Significance, renumbering the other sections accordingly:

2. *Significance.*—This method is intended as a screening test to aid in selecting greases which appear to have promise as ball and roller bearing lubricants for use under the temperature limitations described in the method. The nature of the test is such that close discrimination between lubricants cannot be made.

Test for Water and Sediment in Fuel Oils by Centrifuge (D 1796 - 60 T)

Test for Water in Liquid Petroleum Products by Karl Fischer Reagent (D 1744 - 60 T)

Method for Analysis of Commercial Butane-Butene Mixtures (Gas Chromatography Method) (D 1717 - 60 T)

Method for Calculation of Absolute Viscosity (D 1745 - 60 T)

Test for Luminometer Numbers of Aviation Turbine Fuels (D 1740 - 60 T)

Test for Refractive Index of Viscous Materials (D 1747 - 60 T)

Test for Rust Protection by Metal Preservatives in the Humidity Cabinet (D 1748 - 60 T)

ASTM Tables for Positive Displacement Meter Prover Tanks (D 1750 - 60 T)

The committee withdrew from the report as preprinted the recommendation for publication as tentative of the Method of Test for Autoignition Temperature of Liquid Petroleum Products due to negative votes in the committee. The method has been accepted for publication as information only as indicated below.

Accepted as Tentative, Revisions in:

Test for Water and Sediment by Centrifuge (D 96 - 59 T)

Test for Cone Penetration of Lubricating Grease (D 217 - 52 T)

Spec. for Gasoline (D 439 - 58 T)

Test for Unsulfonated Residue of Petroleum Plant Spray Oils (D 483 - 52 T)

Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 - 59 T)

Method of Calculation for Olefins and Aromatics in Gasoline (D 875 – 53 T)
Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 – 52 T)
Test for Knock Characteristics of Aviation Fuels by the Supercharge Method (D 909 – 59 T)
Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019 – 58 T)
Method for Measuring the Temperature of Petroleum and Petroleum Products (D 1086 – 56 T)
Test for Hydrocarbon Types in Liquid Petroleum Products (Fluorescent Indicator Adsorption (FIA) Method) (D 1319 – 58 T)
Test for Tensile Strength of Paraffin Wax (D 1320 – 57 T)
Test for Needle Penetration of Petroleum Waxes (D 1321 – 57 T)
Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method (D 1656 – 59 T)

Adopted as Standard:

Rec. Practice for Volume Calculations and Corrections in the Measurement of Petroleum and Petroleum Products (D 1087 – 57 T)
Test for Kinematic Viscosity (D 445 – 53 T)

The recommendation in the preprinted committee report regarding Method D 445 was for its revision and continuation as tentative. At the session there was approved the recommendation of Committee D-2 for its revision and adoption as standard subject to letter ballot approval of Committee D-2.

Adopted as Standard, Revisions in:

Test for Melting Point of Petrolatum and Microcrystalline Wax (D 127 – 49)
Test for Sulfur in Petroleum Products and Lubricants (Bomb Method) (D 129 – 58)
Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 – 59)
Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (D 665 – 54)
Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 – 59)
Test for Congealing Point of Pharmaceutical Petrolatums (D 938 – 49)

Accepted for Publication as Information Only:

Test for Effect of Air Supply on Smoke Density from the Burning of Distillate Fuel Oils
Test for Tetraethyllead in Gasoline by Flame Photometer
Test for Naphthenes in Reformer Charge and Product by Refractivity Intercept
Test for Depentanization of Gasoline and Naphthas
Test for the Isolation of Representative Saturates Fraction from Low-Olefinic Petroleum Stocks
Test for Sulfate Content of Oil-Soluble Sulfonates
Centrifuge Method for Preparing Specimen Grids for Use in the Study of Diesel Fuels by Electron Microscopy, including an Electron Micrograph Chart
Recommended Automatic Digital Computer Subroutine, Tables 6 and 7 of ASTM Standard D 1250, Volume Correction Tables
Test for Autoignition Temperature of Liquid Petroleum Products

Committee D-4 on Road and Paving Materials:

Report presented by A. B. Cornthwaite, chairman, and the following actions taken:

Accepted as Tentative:

Test for Effect of Heat and Air on Asphaltic Materials (Thin-Film Oven Test) (D 1754 – 60 T)
Specs. for Preformed Expansion Joint Fillers for Concrete Paving and Structural Construction (Nonextruding and Resilient Bituminous Types) (D 1751 – 60 T)
Specs. for Preformed Expansion Joint Fillers for Concrete Paving and Structural Construction (Nonextruding and Resilient Nonbituminous Types) (D 1752 – 60 T)
Specs. for Hot-Mixed, Hot-Laid Tar Paving Mixtures (D 1753 – 60 T)

Accepted as Tentative, Revisions in:

Test for Resistance to Plastic Flow of Bituminous Mixtures by Means of the Marshall Apparatus (D 1559 – 58 T)
Def. of Terms Relating to Materials for Roads and Pavements (D 8 – 55)

Adopted as Standard:

Test for Compressive Strength of Bituminous Mixtures (D 1074 – 58 T)
Test for Moisture or Volatile Distillates in Bituminous Paving Mixtures (D 1461 – 57 T)

Adopted as Standard, Revisions in:

Methods of Testing Emulsified Asphalts (D 244 – 55)

Standards Withdrawn:

Specs. for Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surface Courses (D 947 – 55)
Specs. for Asphaltic Mixtures for Sheet Asphalt Pavements (D 978 – 54)
Test for Testing Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 – 49)

Committee D-11 on Rubber and Rubber-like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Latex Dipped Goods and Coatings for Automotive Applications (D 1764 – 60 T)

Specs. for Carbon Blacks Used in Rubber Products (D 1756 – 60 T) (Jointly with Committee D-24)

Test for Solubility of Organic Materials (D 1766 – 60 T)

Accepted as Tentative, Revisions in:

Methods for Chemical Analysis of Rubber Products (D 297 – 59 T)

Spec. for Elastomer Compounds for Automotive Applications (D 735 – 59 T)

Methods of Testing Coated Fabrics (D 751 – 59 T)

Test for Accelerated Ozone Cracking of Vulcanized Rubber (D 1149 – 55 T)

Rec. Practice for Description of Types of Styrene-Butadiene Rubbers (SBR) and Butadiene Rubbers (BR) (D 1419 – 60 T)

Methods of Testing Carbon Blacks in Rubber (D 1522 – 58 T)

Adopted as Standard:

Spec. for Insulated Wire and Cable: Vinyl Chloride Plastic Insulating Compound (D 734 – 50 T)

Spec. for Heavy Duty Black Neoprene Sheath for Wire and Cable (D 752 – 57 T)

Spec. for General Purpose Neoprene Sheath for Wire and Cable (D 753 – 57 T)

Spec. for Ozone Resisting Butyl Rubber Insulation for Wire and Cable (D 1352 – 59 T)

Spec. for Synthetic Rubber Heat or Moisture-Resisting Insulation for Wire or Cable (D 1520 – 58 T)

Spec. for Synthetic Rubber Performance, Moisture-Resisting Insulation for Wire and Cable (D 1521 – 58 T)

Spec. for Synthetic Rubber Insulation for Wire and Cable, 90 C Operation (D 1523 – 58 T)

Methods of Testing Flat Rubber Belting (D 378 – 51 T)

Test for Resistance to Surface Cracking of Stretched Rubber Compounds (D 518 – 57 T)

Method of Testing Automotive Air Brake and Vacuum Brake Hose (D 622 – 57 T)

Test for Volume Resistivity of Electrically Conductive Rubber and Rubber-like Materials (D 991 – 48 T)

Test for Evaluating Pressure Sealing Properties of Rubber and Rubber-like Materials (D 1081 – 58 T)

Test for Evaluating Low Temperature Characteristics of Rubber and Rubber-like Materials by a Temperature-Retraction Procedure (TR Test) (D 1329 – 58 T)

Test for Change in Length of an Elastomeric Vulcanizate Resulting from Immersion in a Liquid (D 1460 – 57 T)

Rec. Practice for Standard Test Temperatures for Rubber and Rubber-like Materials (D 1349 – 58 T)

Standard Withdrawn:

Spec. for Natural Rubber AO Insulation for Wire and Cable, 60 C Operation (D 27 – 58)

Tentative Withdrawn:

Methods of Identification and Quantitative Analysis of Synthetic Elastomers (D 833 – 46 T)

The committee withdrew from the preprinted report the following recommendations: Proposed Tentative Specs. for Rubber Rings for Asbestos-Cement Pipe; revision of Tentative Spec. for Nonmetallic Gasket Materials for General Automotive and Aeronautical Purposes (D 1170 – 59 T); adoption as standard of Tentative Method of Test for Resistance to Accelerated Light Aging of Rubber Compounds (D 750 – 55 T), Methods for Chemical Analysis of Synthetic Elastomers (Solid Styrene-Butadiene Copolymers) (D 1416 – 58a T), Methods of Testing Synthetic Rubber Latices (Styrene-Butadiene Copolymers) (D 1417 – 57 T), and Method of Measuring Low-Temperature Stiffening of Rubber and Rubber-Like Materials by Means of a Torsional Wire Apparatus (D 1053 – 58 T).

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials:

Report presented by W. E. Sisco, chairman, and the following actions taken:

Adopted as Standard:

Test for Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration (D 1491 – 57 T)

Test for Bromine Index of Aromatic Hydrocarbons by Coulometric Titration (D 1492 – 57 T)

Adopted as Standard, Revisions in:

Test for Solidification Point of Benzene (D 852 – 47)

Accepted for Publication as Information Only:

Recommendations for Interlaboratory Testing of Industrial Aromatic Hydrocarbons and Related Materials

Committee D-19 on Industrial Water:

Report presented in the absence of the chairman by P. J. Smith, ASTM staff, and the following actions taken:

Accepted as Tentative:

Test for Operating Performance of Cation-Exchange Materials—Sodium Cycle (D 1782 - 60 T)
Test for Phenolic Compounds in Industrial Water and Industrial Waste Water (D 1783 - 60 T)

Accepted as Tentative, Revisions in:

Test for Phosphate in Industrial Water (D 515 - 58 T)
Test for Iron in Industrial Water and Industrial Waste Water (D 1068 - 55 T)

Adopted as Standard:

Test for Identification of Types of Microorganisms and Microscopic Matter in Industrial Water and Industrial Waste Water (D 1128 - 58 T)
Test for Nitrite Ion in Industrial Water (D 1254 - 57 T)
Test for Specific Gravity of Industrial Water and Industrial Waste Water (D 1429 - 56 T)
Test for Dissolved and Gaseous Hydrogen in Industrial Water (D 1588 - 58 T)
Test for Dissolved Oxygen in Industrial Waste Water (D 1589 - 58 T)
Test for Surface Tension of Industrial Water and Industrial Waste Water (D 1590 - 58 T)
Test for Chloroform-Extractable Matter in Industrial Water and Industrial Waste Water (D 1178 - 54 T)
Test for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Industrial Waste Water (D 1252 - 58 T)

Adopted as Standard, Revisions in:

Test for Hardness in Industrial Water (D 1126 - 57)
Def. of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 59)
Test for Oily Matter in Industrial Waste Water (D 1340 - 56)

Committee D-20 on Plastics:

Report presented by F. W. Reinhart, chairman, and the following actions taken:

Accepted as Tentative:

Test for Brittleness Temperature of Plastic Film by Impact (D 1790 - 60 T)
Test for Evaluation of Welding Performance for Poly(Vinyl Chloride) Structures (D 1789 - 60 T)
Spec. for Rigid Acrylonitrile-Butadiene-Styrene (ABS) Plastics (D 1788 - 60 T)
Spec. for Toluenediisocyanate and Poly(Oxypropylene Glycol) (D 1786 - 60 T)
Spec. for IPS Rigid Poly(Vinyl Chloride) (PVC) Plastic Pipe (D 1785 - 60 T)
Spec. for Rigid Poly(Vinyl Chloride) Compounds (D 1784 - 60 T)

Accepted as Tentative, Revisions in:

Test for Tensile Properties of Plastics (D 638 - 58 T)
Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 - 58 T)
Spec. for Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes (D 702 - 59 T)
Spec. for Melamine-Formaldehyde Molding Compounds (D 704 - 56 T)
Def. of Terms Relating to Plastics (D 883 - 59 T)
Test for Measurement of Density of Plastics by the Density-Gradient Technique (D 1505 - 57 T)
Abbreviations of Terms Relating to Plastics (D 1600 - 58 T)
Methods of Testing Urethane Foam Raw Materials (D 1638 - 59 T)
Test for Environmental Stress-Cracking of Type I Ethylene Plastics (D 1693 - 59 T)
Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 - 51) (Standard reverted to tentative)

Adopted as Standard:

Test for Dilute Solution Viscosity of Vinyl Chloride Polymers (D 1243 - 58 T)
Test for Diffuse Light Transmission Factor of Reinforced Plastic Panels (D 1494 - 57 T)
Test for Transverse Load of Corrugated Reinforced Plastic Panels (D 1502 - 57 T)
Test for Bearing Load of Corrugated Plastic Panels (D 1602 - 58 T)
Spec. for Cellulose Acetate Sheet and Film for Primary Insulation (D 1202 - 52 T) (subject to confirming action by Committee D-9)
Spec. for Styrene-Acrylonitrile Copolymer Molding and Extrusion Materials (D 1431 - 56 T)
Spec. for Cellulose Propionate Molding and Extrusion Materials (D 1562 - 58 T)

Standard Withdrawn:

Test for Diffusion of Light by Plastics (D 636 - 54)

Tentatives Withdrawn:

Spec. for Methylstyrene-Acrylonitrile Copoly-

mer Molding and Extrusion Materials (D 1594 - 58 T)
Spec. for Polymethylstyrene Molding and Extrusion Material (D 1595 - 58 T)

The committee withdrew from the report as preprinted the following recommendations: Proposed Tentative Spec. for Styrene-Butadiene Molding and Extrusion Materials; adoption as standard of Tentative Spec. for Biaxially Oriented Styrene Plastics Sheet (D 1463 - 58 T) and Tentative Spec. for Nonrigid Vinyl Chloride Plastic Sheeting (D 1593 - 58 T).

Committee D-27 on Electrical Insulating Liquids and Gases:

Report presented in the absence of the chairman by Charles A. Johnson, secretary, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Estimating Relative Temperature Limits of Dielectric Liquids (D 1805 - 60 T)
Test for Refractive Index and Specific Optical Dispersion of Hydrocarbon Liquids (D 1807 - 60 T)
Test for Refractive Index of Askarels (D 1806 - 60 T)
Method for Sampling Askarels (D 1809 - 60 T)
Test for Volume of Oil in Oil-Contaminated Askarels (D 1808 - 60 T)
Test for Specific Gravity of Askarels (D 1810 - 60 T)
Test for Free Chlorides in Askarels (D 1811 - 60 T)

Adopted as Standard:

Test for 2,6-Ditertiary-Butyl Para-Cresol in New Electrical Insulating Oils (D 1473 - 58 T)
Test for Water in Insulating Liquids (Karl Fischer Method) (D 1533 - 58 T)
Test for Peroxide Number of Mineral Insulating Oils (D 1563 - 58 T)

Adopted as Standard, Revisions in:

Methods of Testing Askarels (D 901 - 56)

Committee E-11 on Quality Control of Materials:

Report presented by Simon Collier, chairman, and the following action taken:

Accepted as Tentative, Revisions in:

Rec. Practices for Designating Significant Places in Specified Limiting Values (E 29 - 58 T)

Joint Committee on Leather:

Report presented in the absence of the chairman by B. L. Lewis, vice-chairman, and the following actions taken:

Accepted as Tentative:

Method for Measuring Thickness of Leather Units (D 1814 - 60 T)
Method for Measuring Thickness of Leather Test Specimens (D 1813 - 60 T)
Test for Water Absorption (Static) of Leather (D 1815 - 60 T)

Accepted as Tentative, Revisions in:

Test for Bond Strength of Leather Belting (D 1699 - 59 T)

Adopted as Standard:

Test for Area of Leather (D 1515 - 57 T)
Test for Width of Leather (D 1516 - 57 T)
Test for Corrosion Produced by Leather in Contact with Metal (D 1611 - 58 T)
Method of Conditioning Leather and Leather Products for Testing (D 1610 - 58 T)
Def. of Terms Relating to Leather (D 1517 - 57 T)

The committee withdrew from the preprinted report the proposed Tentative Method of Test for Cold Crack Resistance of Upholstery Leather.

THIRTY-SEVENTH SESSION—COMMITTEE REPORT SESSION

FRIDAY, JULY 1, 12:30 P.M.

SESSION CHAIRMAN: A. C. WEBBER

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by R. A. Harris, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings (B 361 - 60 T)

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Sand Castings (B 26 - 59 T)
Spec. for Aluminum-Alloy Sheet and Plate (B 209 - 59 T)

Spec. for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 59 T)
Spec. for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 59 T)
Spec. for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 59 T)
Spec. for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 59 T)
Spec. for Aluminum-Alloy Extruded Tubes (B 235 – 59 T)
Spec. for Aluminum-Alloy Pipe (B 241 – 59 T)
Spec. for Aluminum-Alloy Die Forgings (B 247 – 59 T)
Spec. for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 – 59 T)
Spec. for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 – 59 T)
Spec. for Aluminum-Alloy Round Welded Tubes (B 313 – 59 T)
Spec. for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 – 59 T)
Spec. for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 – 59 T)
Spec. for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 – 59 T)
Spec. for Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 – 59 T)

Adopted as Standard:

Spec. for Aluminum Alloy Hardeners Used in Making Zinc-Base Die-Casting Alloys (B 327 - 58 T)

Adopted as Standard, Revisions in:

Spec. for Aluminum for Use in Iron and Steel Manufacture (B 37 – 59)
Spec. for Magnesium-Base Alloy Sheet and Plate (B 90 – 59)
Spec. for Magnesium-Base Alloy Forgings (B 91 – 59)
Spec. for Magnesium Ingot and Stick for Remelting (B 92 – 57)
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 59)
Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 59)
Spec. for Magnesium-Base Alloy Extruded Tubes (B 217 – 59)

Committee C-1 on Cement:

Report presented in the absence of the chairman by W. S. Weaver, vice-chairman, and the following actions taken:

Accepted as Tentative:

Test for False Set of Portland Cement (Paste Method) (C 451 – 60 T)
Test for Potential Sulfate Resistance of Portland Cement (C 452 – 60 T)

Accepted as Tentative, Revisions in:

Methods of Sampling Hydraulic Cement (C 183 – 58 T)
Spec. for Portland Blast-Furnace Slag Cement (C 205 – 58 T)
Test for Fineness of Hydraulic Cement by the No. 325 Sieve (C 430 – 59 T)

Adopted as Standard, Revisions in:

Spec. for Masonry Cement (C 91 – 59)
Spec. for Portland Cement (C 150 – 59)
Test for Autoclave Expansion of Portland Cement (C 151 – 59)
Spec. for Air-Entraining Portland Cement (C 175 – 59)
Def. of Terms Relating to Hydraulic Cement (C 219 – 55)

Committee C-11 on Gypsum:

Report presented in the absence of the chairman by R. H. Faber, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Annular Ringed Nails for Gypsum Wallboard (C 380 – 58 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Gypsum (C 11 – 59)
Methods of Testing Gypsum and Gypsum Products (C 26 – 59)
Spec. for Gypsum Plasters (C 28 – 59)
Spec. for Gypsum Wallboard (C 36 – 58)

Committee C-15 on Manufactured Masonry Units:

Report presented in the absence of the chairman by P. M. Woodworth, vice-chairman, and the following actions taken:

Adopted as Standard:

Specs. for Industrial Floor Brick (C 410 – 57 T)

Adopted as Standard, Revisions in:

Methods of Sampling and Testing Brick (C 67 – 57)
Methods of Sampling and Testing Structural Clay Tile (C 112 – 52)
Spec. for Structural Clay Facing Tile (C 212 – 54)
Spec. for Facing Brick (Solid Masonry Units Made from Clay or Shale) (C 216 – 57)

Committee E-2 on Emission Spectroscopy:

Report presented by D. L. Fry, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practices for Spectrochemical Computations (E 158 – 60 T)

Accepted for Publication as Information Only:

Rec. Practice for Installation and Safe Operation of the Spectrochemical Laboratory
Rec. Practice for the Use of Statistical Methods in Spectrochemical Methods
Rec. Practice for Flame Photometric Methods of Spectrochemical Analysis
Rec. Practice for Establishing and Controlling Spectrochemical Analytical Curves
Method for Spectrochemical Analysis of Beryllium Copper Alloys by the Rotating Disk Spark Technique
Method for Spectrochemical Analysis of Zinc and Its Alloys by the Point-to-Plane Spark and Condensed Arc Techniques Using a Recording Photoelectric Spectrometer
Method for Spectrochemical Analysis of Lead and Its Alloys by the Point-to-Plane Technique Using a Recording Photoelectric Spectrometer
Method for Spectrochemical Analysis of Cast Iron by the Point-to-Plane Spark Technique
Method for Spectrochemical Analysis of Plain-Carbon and Low-Alloy Steel by the Point-to-Plane Method
Method for Spectrochemical Analysis of Plain-Carbon and Low-Alloy Steel by the Rod-to-Rod Spark Technique
Method for Spectrochemical Analysis of Stainless Type Steels by the Point-to-Plane Spark Technique
Method for Spectrochemical Analysis of Steel by the Solution-Rotating Disk-Spark Technique
Method for Spectrochemical Analysis of Cast Iron for Cerium by the Point-to-Plane Condensed Arc Method
Method for Spectrochemical Analysis of Plain Carbon and Low Alloy Steel by the Pellet Technique
Method for Spectrochemical Analysis of Cement for Manganic, Sodium, and Potassium Oxides by the Flame Photometer Technique
Method for Spectrochemical Analysis of Glass for Alkaline Earth Elements by the Flame Photometer Technique

Committee E-7 on Nondestructive Testing:

Report presented by J. H. Bly, chairman, and the following actions taken:

Accepted as Tentative:

Methods for Liquid Penetrant Inspection (E 165 – 60 T)
Method for Ultrasonic Contact Inspection of Weldments (E 164 – 60 T)

Committee E-15 on Analysis and Testing of Industrial Chemicals:

Report presented by W. A. Kirklin, chairman, and the following action taken:

Accepted for Publication as Information Only:

Rec. Practice for Developing Precision Data on ASTM Methods for the Analysis and Testing of Industrial Chemicals

14TH EXHIBIT OF TESTING AND SCIENTIFIC APPARATUS AND LABORATORY SUPPLIES

MONDAY, JUNE 27 TO FRIDAY, JULY 1

The biennial Exhibit of Testing and Scientific Apparatus Laboratory Supplies was an added educational feature of the Annual Meeting. Forty-nine exhibitors took part. In addition to those registered for the Annual Meeting, 332 went through the exhibit.

1960 PHOTOGRAPHIC EXHIBIT

The biennial Photographic Exhibit included the display of 233 entries relating to research and testing techniques and photomicrography.

EDITOR'S NOTE.—The recommendations affecting standards referred to letter ballot were approved when the ballot was canvassed on September 19.

INTERNATIONAL ASPECTS OF ASTM

ANNUAL ADDRESS BY THE PRESIDENT

BY F. L. LAQUE[1]

In dealing with some international aspects of ASTM, it is difficult to resist the temptation to describe such activities by numerous references to statistical data.

For example, we have over 1400 members from over 50 foreign countries. Our income from sales of our publications abroad amounted to over $200,000 in 1959. This resulted from over 9000 orders from 65 countries ranging alphabetically from Afghanistan to the Windward Islands.

In collaboration with the American Standards Assn. we have contributed substantially to the work of the International Standards Organization. Several technical committees cooperate continually with corresponding technical organizations elsewhere in the world, for example, the activities of Committee D-2 on Petroleum Products and Lubricants in collaboration with the Institute of Petroleum (London).

Numerous other examples with supporting statistical data could be cited, but since information of this sort tends to be about as dull as it is impressive, I shall assume that such documentation is unnecessary in this instance. I feel that most people recognize that ASTM operates in a most substantial way on an international scale and occupies a most important position among societies having an international scope.

Any who may seek more detailed support of the statements just made can secure a more than ample documentation by communication with the Staff, or they can get in touch with me directly, since the Staff has furnished me with an abundance of statistical information and examples of international activities for possible use in composing these remarks.

I have suggested to the Headquaters Staff that they prepare a general account of ASTM activities on an international scale for publication in some future issue of the ASTM BULLETIN.

What I wish to concentrate on are two principal advantages of ASTM standards as they affect our relations with other countries. In each case, the advantage is reciprocal. It helps us and it helps the other countries.

Our government is committed to a policy of providing technical assistance to other countries, and particularly to what are called "underdeveloped" countries. It will be readily recognized that in the field of materials, the 14,359 pages in the ten volumes of ASTM standards comprise the greatest concentration of immediately and practically usable information that it would be possible to furnish anyone needing such data. The materials specifications and the methods of test by which properties of materials are appraised represent a sort of distillation product from the tremendous amount of materials research that has been carried out over the years by ASTM or under ASTM auspices. In most cases, no further work or search for data should be required to characterize a material needed for some specific purpose whether it be concrete for a wall, steel for a bridge, or fuel for an engine. The methods of test that are described provide not only a basis for inspection of what is furnished, but indicate, as well, the paths that may be followed in the search for new and improved materials.

---

[1] Vice-President, Manager of Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

It should be evident, therefore, that through its work on standards, the ASTM has made a unique contribution to the ability of this country to lend technical assistance to other countries.

A second value of ASTM standards is related to their use by foreign purchasers in connection with the procurement of goods made in this country and furnished in accordance with ASTM standards. The first value is to the purchaser who finds an ASTM standard to be the best available way for him to describe what he wants and to be sure he gets what he has asked for. This latter aspect takes the form of evidence of compliance with the standard based on a precise means of inspection.

Such use of ASTM standards in the purchase of goods made in this country is of obvious benefit to domestic producers in increasing the volume of their export business. It provides a ready means whereby the extra value associated with the quality and precision represented by ASTM standards can be described to a prospective purchaser in a way that he can recognize. They also afford assurance that the required qualities will be delivered. A greater use of ASTM standards in foreign trade should go a long way toward improving the demand for such high-quality materials and in counteracting the dubious economic advantage of lower-grade materials of uncertain and undefined qualities. Such lower-grade materials usually are offered from sources able to quote lower prices based on low-cost labor working to undefined standards.

It seems safe to conclude that U. S. manufacturers are not likely to be able to compete with the products of cheap foreign labor on a price basis. In competing on the basis of superior qualities that can be described, assured, and measured by reliable means, nothing will be more helpful than the general use of ASTM standards as a basis for offer and purchase.

Industry in this country should cultivate the advantage offered to them by the use of ASTM standards abroad by identifying its products with these standards to the fullest possible extent and by cultivating the use of such standards by foreign purchasers of their products.

For the reasons just described, it is most essential that steps be taken to make ASTM standards more readily usable outside this country. This will require translation of the standards into other languages. A necessary first step in each case would be the preparation of a glossary of terms in the language of the proposed translation to take into account the pertinent idiom as well as the formal language of the other country. Presumably, such translations and their publication would have to be undertaken in appropriate stages, determined by priority of interests in particular areas.

A start along the lines indicated has been made with special reference to Latin America. We have had conversations with officials of the U. S. Departments of Commerce and State and have reason to hope that considerable help will come from these sources. For its part, the ASTM has offered these governmental departments the free use of its standards material and its full cooperation in making ASTM standards more readily usable in other countries.

Special efforts have been made over the years to assist Latin American countries in the organization of standardization activities. For example, in 1957, Dr. Bates and Mr. Painter participated in an Inter-American Standards meeting held in Rio de Janeiro, under the auspices of the Inter-American Economic and Social Council of the Organization of American States. More recently, there has been some renewed activity through the International Cooperation Administration. Through these and other appropriate channels, it is hoped that further progress can be made toward standardization and the use of ASTM standards in Latin America. Many of us believe that aside from the technical and industrial benefits arising from the use of ASTM research and standards, much good will is generated. This could be a most important by-product.

I recommend to my successors that they make a most diligent effort to pursue this matter on a broad international scale and thereby insure that this aspect of ASTM activities in the international field will be of maximum benefit to our foreign friends, to American industry, and to the Society itself.

1960 ANNUAL REPORT OF THE BOARD OF DIRECTORS

	Page
Technical Activities	41
National and Other Meetings	45
Publications	46
District and Related Activities	46
Membership	47
Honors and Awards	50
Finances	51
Administrative Matters	53
Division of Materials Sciences	54
Cooperative Activities	54
Long-Range Planning Committee	56
Appendix I—Report of Auditors	57
Reports of Administrative Committees:	
Appendix II—on Standards	71
Appendix III—on Research	78
Appendix IV—on Papers and Publications	79
Appendix V—on District Activities	84
Appendix VI—on Simulated Service Testing	85
Appendix VII—on Education in Materials	86

In this, the Board's report for 1960, the year's activities are reviewed in the light of its responsibilities to see that the purposes for which the Society was organized are being carried out. These purposes are implemented through the activities of the technical committees, the standards publications and compilations of data, the sponsoring of meetings and discussions, and the publication of technical papers. These are all discussed in later sections.

It has been a noteworthy year in many respects. Of especial significance has been the creation of committees in new fields—the sampling and analysis of metallic ores, the study of skid resistance of traffic surfaces, standardization work in the field of ceramics for electronics, and the exploration of sensory testing. Important, too, was the holding of two national meetings—the Annual Meeting last June, which was the largest meeting yet held in point of attendance, and the Pacific Area National Meeting held in San Francisco in October, which was the largest meeting in number of sessions and technical papers. These two meetings are reflected in the number of Special Technical Publications that resulted. The importance of these, of course, is the value of the subject matter covered and the timeliness of the presentations. Every effort is being made to publish these books promptly.

During the year, the new Division of Materials Sciences took shape. The Division is sponsoring two symposia of fundamental significance at this Annual Meeting: on Recent Progress in Materials Sciences, and on Nature and Origin of Strength of Materials. It is also planned to effect formal organization shortly so as to place the Division on a permanent working basis, and in so doing to tie in the work of the technical committees effectively in the activities sponsored by the new Division.

The membership record is an indication of the extent to which those interested are being reached and enrolled in the Society's activities (present membership, 10,263). A review of the financial operations gives an indication of how well the Society is equipped to handle the continuing expansion in activities, including Headquarters needs to house any necessarily increased Staff.

A development of prime significance was the fulfillment of the Board's plan to establish a fellowship at a school of engineering and grants-in-aid at several others.

Technical Activities

WHILE WE customarily think of technical committee activities in terms of standards, a number of the committees are responsible for thoroughgoing research programs that yield many valuable data. Worthy of special mention is the final report on the testing of full-size wood poles and the preliminary report of the special committee on fracture testing of sheet materials. Practically the entire West Coast meeting consisted of technical programs sponsored by the technical committees.

The standards activities for the year are reviewed in the report of the Administrative Committee on Standards appended hereto.[1]

Apart from the organization of new committees mentioned later, a number of enlargements or refinements of scopes of existing committees were approved, as described herein.

Revised Committee Scopes, Titles, and Jurisdictions:

Committee B-4.—Further studies in Committee B-4 since the last report have resulted in a decision to continue to limit its scope to metallic materials; but in order to make the committee's title more descriptive of its activities, the title has been revised as follows by the addition of the word "Thermostats":

> "Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating, and Contacts."

Committee B-8.—On the recommendation of Committee B-7 on Light Metals and Alloys, its Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium has been transferred to the jurisdiction of Committee B-8 on Electrodeposited Metallic Coatings. The title of Committee B-8 has been revised as follows by the addition of the words "and Related Finishes":

> "Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes."

The scope of Committee B-8 has been revised as follows by the omission of the words in brackets and the addition of the italicized words:

> "To prepare specifications for electrodeposited, *anodized*, and chemically reduced [metallic] coatings of all kinds on all types of basis material, subsequent treatments of such coatings, methods of testing these coatings and materials required for their production; including all pertinent investigations of the substrates, the methods of production, the properties of these [deposits] *coatings* and their performance under conditions of use and test; excluding only specifications for metal products to which such coatings are applied."

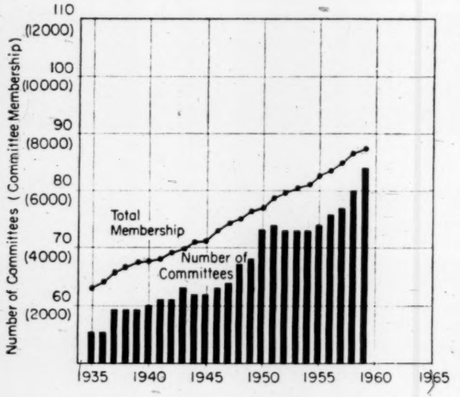
Committee C-3.—A suggested expansion of scope of Committee C-3 on Chemical-Resistant Mortars to include membranes and linings is under consideration, and a study committee has been appointed to recommend a revised title and scope. Such expansion might include monolithic floor surfacing compounds, troweled-on tank linings, bed joints, grouts, and latex-cement mortar.

---

[1] See p. 71.

Committee D-14.—To define its present activities more precisely, the scope of Committee D-14 on Adhesives has been revised from: "The formulation of specifications, methods of test, and definitions of terms pertaining to adhesives, including animal, vegetable, mineral, and synthetic types," to read as follows:

> "The development of methods of testing, specifications, definitions of terms, and recommended practices for adhesives and materials used in compounding adhesives and research on adhesives and adhesion, including the clarification of the nature of adhesion. Standards for compounding ingredients, when needed, will be developed by Committee D-14 when not available from other ASTM committees, with suitable coordination."



Showing Growth in Number of Committees and in Official Committee Membership.

Committee C-16.—The Board has approved the addition of the italicized words to the scope of Committee C-16 on Thermal Insulating Materials:

> "Formulation of test methods and specifications relating to thermal insulating materials (except those used in the insulating refractory field) and any protective coating material *and to coverings* associated therewith to preserve their thermal insulating efficiency."

New Activities:

Joint Sealants.—At its May, 1959, meeting the Board authorized the organization of a new technical committee to cover elastomeric caulking and sealing compounds. The organization meeting was held at Society Headquarters on September 15, 1959; the title of the new committee is Committee C-24 on Joint Sealants, and its scope is as follows:

> "The promotion of knowledge, stimulation of research and the development of methods of test, specifications and nomenclature of materials for sealing joints and openings. Such sealants include caulking and glazing compounds and shapes. These activities will be coordinated with those of other relevant committees of ASTM.
>
> "The committee is not concerned with oil-base putty, glazing and caulking compounds now under the jurisdiction of Committee D-1 on Paint, Varnish, Lacquer and Related Products."

Ore Sampling and Analysis.—On the recommendation of a conference held in June, 1959, a new Committee E-16 on Sampling and Analysis of Metal-Bearing Ores was authorized. The committee was formally organized at a meeting held at Society Headquarters on January 13, 1960, and the following statement of scope has been approved:

> "The promotion of knowledge of and the formulation of methods of sampling, analysis, and testing of natural and processed metal-bearing ores and related raw materials such as fluxes. This activity is to be coordinated with the work of relevant committees in ASTM and other organizations."

Skid Resistance.—On the recommendation of a conference of representatives of technical committees held during the 1959 Annual Meeting, the Board authorized the formation of a new technical committee to undertake work on skid resistance properties. While inclusion of surface roughness and riding characteristics was discussed, it was thought desirable to confine the immediate efforts to skid resistance. The following statement of scope, recommended by a steering committee at a meeting held at Society Headquarters on December 16, 1959, has been approved in principle by the Board:

"(*a*) To develop and standardize field and laboratory methods of test for determining traffic surface slipperiness,

"(*b*) To develop and standardize methods of test for the use of a set of standards of traffic surface slipperiness,

"(*c*) To stimulate research to accomplish the foregoing purposes."

Formal organization of the committee will be effected at a meeting on June 28 during the Annual Meeting. The committee's title is Committee E-17 on Skid Resistance.

Electronic Ceramics.—Reference was made in the report a year ago to the activities of the American Ceramic Society and to the interest of several ASTM committees in the subject of electronic ceramics. Since then the Electronics Division of the American Ceramic Society has requested the Society to organize a new technical committee to deal with standards in this field. The Board appointed a steering committee to look into the matter, and on its recommendation a new Committee C-25 on Ceramics for Electronics has been authorized, with limitations in its scope that would recognize the interests of other committees, in particular Committees D-9 on Electrical Insulating Materials and F-1 on Materials for Electron Tubes and Semiconductor Devices. The following statement of scope recommended by the committee at its organization meeting on April 28 has been approved:

"Definitions of terms and formulation of test methods and specifications covering ceramic materials for electronic applications and promotion of research in this field.

"The principal materials included in this scope are: low-loss dielectric ceramics, ferroelectric and nonmetallic magnetic materials, and ceramic-type semiconductors and composites. The scope does not include materials for electron tubes, electric discharge and semiconductor devices, and electrical tests for electrical porcelain."

Sensory Testing.—Mention was made in the report a year ago of a recommendation that the Society evaluate the desirability of organizing a committee on so-called psychophysical testing. As a result of a very thoroughgoing study, including the holding of a fairly extensive conference of the interested individuals, which resulted in a positive recommendation, the Board has authorized the organization of a new Committee E-18 on Sensory Evaluation of Materials and Products, with the following statement of scope:

"The promotion of knowledge, stimulation of research, and the development of recommended principles and practices for the sensory evaluation (including discrimination and preference) of materials and products."

In taking this action the Board is cognizant of the interest of a number of ASTM committees in some aspects of the subject, and in amplification of the proposed scope of activities the following is quoted from the recommendations of the study committee as representing the ways in which the proposed committee should function:

1. Cooperation with other committees of the Society and with other organizations.
2. Preparation of standard definitions and nomenclature.
3. Establishment of principles of psychophysical test methods.
4. Preparation of recommended practices for design and conduct of tests, collection of

data, and analysis and interpretation of results.

5. Fostering study of relationships between sensory evaluations and physical and chemical measurements.

6. Dissemination of information by technical publications and symposia.

Graphite.—Supplementing the reference in last year's report to suggested work on graphite for nuclear use, Subcommittee V on Nuclear Applications of Committee C-21 on Ceramic Whitewares and Related Products has been authorized to proceed with the formation of a task group to handle this activity. There is a good nucleus of qualified men in the subcommittee, and it is the intention to take advantage of such qualified participants in the selection of the task group.

Heavy Water.—The Society has been asked to prepare specifications for heavy water for nuclear applications. The Society's Committee D-19 on Industrial Water is prepared to handle the preparation of such specifications, but its official scope is limited to methods of testing, and the committee is not classified according to producers, consumers, and general interests. The Board has authorized Committee D-19 to assign development of these specifications to a task group with balanced representation, with the understanding that in conducting the ballot on the approval of the specifications in the entire Committee D-19 the vote on the specifications will be classified.

Gall and Wear Testing.—As the result of an announcement in the ASTM BULLETIN inquiring whether there would be interest in the subject of gall and wear testing, considerable interest was expressed, in view of which a conference of those concerned will be held to determine the desirability of organizing a committee on the subject.

Cement and Concrete Reference Laboratory:

For a number of years Committee C-1 on Cement has sponsored at the National Bureau of Standards, with its cooperation, a Cement Reference Laboratory for the purpose of evaluating testing equipment in cement testing laboratories throughout the country. This is now being extended to include concrete testing as well as cement testing, and the work will be under the joint auspices of Committees C-1 and C-9 on Concrete and Concrete Aggregates. A joint committee is being set up to be responsible for this work. The Board has formally approved this activity.

Product Specifications:

Recently the Society has been asked to expand its work dealing with product specifications. One of these requests, for specifications for plastics used in building construction, has come from the Building Research Advisory Board, and the Federal Housing Administration. Work is already under way on plastic pipe, but the coverage that is desired will be much broader than this. Another request is for specifications for aluminum guard rails for highways.

The Society, of course, has developed a number of product specifications in various fields, such as insulated wire and various steel products. In each instance it has been left to the technical committees to evaluate the need for and utility of such specifications, having in mind primarily that the specifications should cover an item that would be generally available and not be of a special nature. The present demand for the extension of the Society's work on product specifications is a very significant one indeed and will need to be given careful study.

National and Other Meetings

1959 Annual Meeting:

The Sixty-second Annual Meeting of ASTM in Atlantic City, N. J., June 21 to 26, 1959, again broke all records, with a total registration of 3133. Over 900 meetings of technical committees were held. The program consisted of 36 technical sessions, two special luncheons, and the Annual Dinner. The committee reports presented, as well as a number of the technical papers, are to be found in the 1959 *Proceedings*. Other papers, particularly those comprising symposia, have appeared in various issues of the ASTM BULLETIN and in separate special technical publications (STP's). A review of the meeting appeared in the July, 1959, ASTM BULLETIN.

District Support.—The Philadelphia District again played host for the Annual Meeting in Atlantic City. With the greater proportion of national meetings being held in Atlantic City, the Society is unusually indebted to the Philadelphia District for its support. The District Council's efforts were outstanding in providing the very excellent ladies' entertainment. Again, a most welcome break in the week of intensive committee meetings was provided by the Philadelphia District in the form of a cocktail party, entertainment, and dinner on Wednesday evening. The efforts of the Philadelphia District are greatly appreciated.

1960 Committee Week:

The Chicago District Council acted as host to Committee Week, and on Wednesday, as a breather, the Council sponsored a very interesting cocktail party and dinner. Following the dinner, Mr. John Nash Ott, famous for his work in time lapse photography, presented a very interesting talk on the various aspects of his work, ranging from nature movies made in cooperation with Walt Disney to films of a highly scientific and fundamental character taken at high magnifications.

The Dinner provided an opportunity to make note of our increase in membership through the presentation by the President of certificates to the 1000th, 5000th and 10,000th member.

During the week, nearly 1000 meetings of committees were held, including an open meeting of Committee E-12 on Appearance constituting a round table discussion on Measurement of Color and Gloss of Anodized Aluminum Alloys.

Pacific Area National Meeting:

The most extensive series of technical sessions ever sponsored by the Society was held during the Third Pacific Area National Meeting in San Francisco, Calif., during the week of October 11–16, 1959—52 technical sessions in all. Five Industry Luncheons and a President's Luncheon were also held during the week. In addition to the President's talk, the President's National Luncheon was addressed by Lt. Gen. Bernard A. Schriever, Commander of Air Research and Development, who emphasized the need for more intensive work on newer materials. Both the President's talk and that of General Schriever appeared in the ASTM BULLETIN.

Along with the extensive sessions and luncheons, over 150 technical committee meetings were held throughout the week, plus a most interesting Exhibit of Testing and Scientific Apparatus and Laboratory Supplies. In all, a most memorable national meeting was held in San Francisco, and the Society is greatly indebted to the San Francisco General Committee on Arrangements for its intensive efforts in connection with the Third Pacific Area National Meeting.

Future Meetings:

Annual Meetings of the Society have been scheduled as follows:

June 25–30, 1961—Atlantic City, N. J.
June 24–29, 1962—New York, N. Y.
June 23–28, 1963—Atlantic City, N. J.
June 21–26, 1964—Chicago, Ill.
June 13–18, 1965—Purdue University, Lafayette, Ind.

Committee Week and Spring Meetings are as follows:

January 29–February 3, 1961—Cincinnati, Ohio
February 5–9, 1962—Dallas, Tex.
February 3–8, 1963—Montreal, Quebec, Canada
February 3–7, 1964—Philadelphia, Pa.
February 8–12, 1965—Cleveland, Ohio

Pacific Area National Meetings are scheduled as follows:

September 30–October 5, 1962—Los Angeles, Calif.
October 10–15, 1965—San Francisco, Calif.

Some consideration is being given to having more frequent meetings in the West Coast Area.

Publications

A complete review of the Society's publications during the past year is set forth in the appended report of the Administrative Committee on Papers and Publications.[2] Apart from this, however, attention is called to a decision of the Board authorizing that the ASTM BULLETIN, beginning in 1961, be issued monthly instead of eight times per year as at present. This decision was reached to provide a more frequent means of bringing news of the Society to the attention of its members. It will also make possible the publication of more technical articles of a feature type which should be of timely interest to the members. The Board also authorized issuing the Book of Standards in eleven rather than in the present ten parts; Part 9 had become too unwieldy, and is being split into two parts.

One development of considerable significance has been the compilation of several symposia and independent technical papers in single STP's (Special Technical Publications) on a given subject. In a sense these represent the proceedings of the Society for the year in a given field. This modification should be of considerable assistance in the search of library material on a single subject.

District and Related Activities

ASTM has 17 districts throughout the United States and part of Canada. These districts, administered by local councils, provide members with the opportunity to take part in local and national ASTM activities, to meet for discussion of technical matters, to hear leading authorities in various fields of technology, and to become acquainted with the officers and other members of ASTM.

During the year the Student Membership Award program administered by districts continued to expand. Of the 17 districts, 13 now have programs in operation which presented 581 awards during the year. Also, 200 additional awards were sponsored by the Board of Directors.

President LaQue spoke before ten districts. Vice-President Bates gave three addresses, while Vice-President Clair spoke at one district meeting. In all, 25 meetings were held, of which 16 were jointly sponsored or were held in cooperation with other technical or educational groups. The accompanying list shows the date and place of each district meeting. Complete news accounts of the meetings were reported throughout the year in the ASTM BULLETIN.

---

[2] See p. 79.

The Rocky Mountain and New England Districts both sponsored all-day sessions which were highly successful. The Philadelphia and Chicago Districts were active as hosts for the 1959 Annual Meeting and 1960 Committee Week Meeting, respectively, and the Southern and Northern California Districts joined in making arrangements for the Third Pacific Area National Meeting in San Francisco.

Districts continued to be active in local technical councils, Engineering Week celebrations, science fairs, etc.

The Administrative Committee on District Activities is investigating the possibility of establishing a new Northwest District to encompass Idaho, Oregon, Washington, and British Columbia.

District Meetings:

St. Louis (jointly with Engineers Club of St. Louis)	October 1, 1959	St. Louis, Mo.
Rocky Mountain (jointly with CSI)	October 20, 1959	U. S. Air Force Academy, Colo.
New York	October 22, 1959	New York, N. Y.
Pittsburgh (jointly with SAE)	October 27, 1959	Pittsburgh, Pa.
New England (jointly with AIChE)	November 12, 1959	Boston, Mass.
Washington	November 12, 1959	Richmond, Va.
Central New York	November 18, 1959	Schenectady, N. Y.
Southeast (jointly with NACE, ASM)	November 18, 1959	University of Tennessee, Knoxville, Tenn.
Detroit	November 19, 1959	Detroit, Mich.
Philadelphia	November 19, 1959	Lafayette, Easton, Pa.
Southeast (jointly with NACE, ASTM Committee D-19)	January 26, 1960	Atlanta, Ga.
New York	January 27, 1960	New York, N. Y.
Southwest	February 8, 1960	University of Houston, Houston, Tex.
Southwest (jointly with NACE, ASM)	February 9, 1960	Houston, Tex.
Southwest (jointly with ASM, NACE)	February 11, 1960	Dallas, Tex.
Washington (jointly with ASTM Committee D-1)	February 16, 1960	Washington, D. C.
Philadelphia (jointly with Engineers Council)	February 24, 1960	Philadelphia, Pa.
Northern California	March 3, 1960	San Francisco, Calif.
Ohio Valley (jointly with ASTM Committees D-9, D-20)	March 9, 1960	Cincinnati, Ohio
Cleveland (jointly with SAE, ASM)	March 30, 1960	Cleveland, Ohio
Pittsburgh	April 13, 1960	Pittsburgh, Pa.
Western New York (jointly with ACS)	April 19, 1960	Niagara Falls, N. Y.
Detroit (jointly with NACE, Electrochemical Society)	April 26, 1960	Detroit, Mich.
Central New York (jointly with FPRS)	April 27, 1960	Syracuse, N. Y.
New England (jointly with Worcester Polytechnic Inst.)	May 6, 1960	Worcester Polytechnic Inst., Worcester, Mass.

Membership

During the year the Society passed its 10,000th member mark. Cognizance of this was taken at the Committee Week dinner by presenting a special certificate to the 10,000th member, Mr. Robert J. McCallin, Technical Service Supervisor of the Medusa Portland Cement Co., Wampum, Pa. Certificates also were given to the 1000th member, Mr. Albert J. Loepsinger, Director of Research, Grinnell Corp., Providence, R. I., who has been a member of the Society since 1908; and to the 5000th member, Mr. Harold Allen, U. S. Bureau of

Public Roads, Washington, D. C., who became a member in 1943.

On May 1, 1960, the membership of the Society totaled 10,263, compared with a total of 9786 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	May 1, 1959	May 1, 1960	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	26	27			2		3		2	3	1
Perpetuity and Life	13	13									
Sustaining	406	407	4			5	6	4	9	10	1
Company, Firm, etc.	2076	2 094	77	22		22	8	131	121	139	18
Individual, etc	6961	7 364	318	135	41	6	76	827	500	903	403
Associate	304	358	14	10		60		138	84	138	54
Total	9786	10 263	413	167	43	93	93	1100	716	1193	477
Student	1484	1 637	151	671		62		1037	884	1037	153

It is interesting to compare our present membership of 10,263 with the figures for ten and twenty years ago; in 1950 our total membership was 6761, and in 1940 it was 4314.

In 1959 more new members were reported than in the previous high year, 1958—1100 compared to 1043. The average in several previous years was from 602 to 775. Our net gain in 1959 was 477.

Associate and Student Memberships:

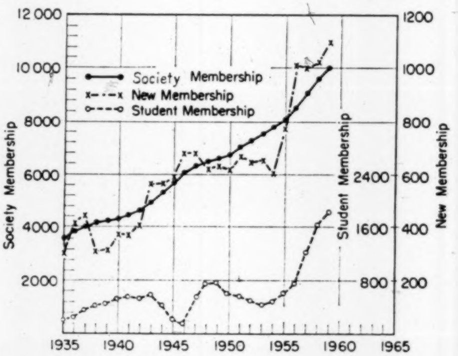
The class of Associate Membership, which extends to 30 years of age and replaced the former junior membership with its 27-year age limit, continues to grow as noted in the table.

Because of the influence of student membership prize awards, which recognize outstanding students in courses related to the Society's work, the very encouraging growth in this class has continued. The Society itself and many of the districts, in addition to individuals and companies, are sponsoring student membership awards.

Sustaining Members:

There have been ten new sustaining members in the period from May, 1959, to May, 1960, and 15 transfers to company memberships. The new sustaining members are:

Acta Technological Laboratories
Atomics International, A Division of North American Aviation, Inc.
Autonetics, A Division of North American Aviation, Inc.
The Glidden Co.
Industrial Fasteners Inst.



Showing Growth in Membership and Acquisition of New Members.

NIBCO, Inc.
North American Aviation, Inc., Engineering Technical Library
North American Aviation, Inc., Missile Development Div., Missile Division Laboratory
Olin Mathieson Chemical Corp., Chemical Div.
Western Electric Co., Manufacturing Div.

50-Year Members:

There are seven individuals and eleven organizations as noted below that this year have been connected with the Society continuously for 50 years:

Charles V. Bacon
P. H. Bates
A. T. Goldbeck
A. E. Horn
William H. Klein
M. E. McDonnell
G. H. Woodroffe

American Transit Assn.
Armco Steel Corp.
The British Ceramic Society
Cementos Mexicanos, S. A.
General Electric Co., Pittsfield Works
Iowa, State University of, Libraries
National Crushed Stone Assn., Inc.
St. Louis, City of, Municipal Testing Laboratory
Stanford University Library
The Studebaker-Packard Corp.
The Western Union Telegraph Co.

These members will be recognized at the Annual Meeting.

40-Year Members:

There are 54 members who this year have completed 40 years of membership, bringing the total number of certificates issued to the "Forty-Year Club" to 516. The members who will receive the 40-Year Certificates at this Annual Meeting are as follows:

American Bridge Division, United States Steel Corp.
American Hard Rubber Co.
O. H. Ammann
Alfred J. Amsler and Co.
The Anaconda Co.
California State Department of Public Works, Division of Architecture
Chase Brass and Copper Co., Inc.
Cities Service Oil Co.
Connecticut State Highway Dept.
Consolidated Edison Company of New York, Inc.
Cook Paint and Varnish Co.
Cornell University, School of Chemical Engineering
Ray B. Crepps
Herbert A. Davis
Drexel Institute of Technology
John R. Dwyer
J. V. Emmons
John R. Freeman, Jr.
General Steel Castings Corp.
James P. Gill
L. T. Holt
Idaho State Department of Highways
D. W. Kessler
Thomas C. Law
Louisville Cement Co.
The Mansfield Tire and Rubber Co.
L. J. Markwardt
Massachusetts Institute of Technology
Minnesota Department of Highways
Mississippi State Highway Dept.
Norman L. Mochel
Benjamin Moore and Co.
R. R. Moore
National Malleable and Steel Castings Co.
New Hampshire Department of Public Works and Highways
New York City, Department of Purchase
Gershon L. Oliensis
Omaha, City of, Department of Public Works
Arthur Phillips
Pittsburgh Plate Glass Co.
The Richardson Co.
Riverton Lime and Stone Co., Inc.
Saskatchewan, University of, Library
Sun Shipbuilding and Dry Dock Co.
Sydney, University of, Fisher Library
Union Carbide Metals Co., Division of Union Carbide Corp.
Universal-Cyclops Steel Corp.
Virginia, Commonwealth of, Department of Highways
Stanton Walker
E. H. Wellemeyer
The West Virginia State Road Commission
Western Brass Mills, Metals Division of Olin Mathieson Chemical Corp.
The S. S. White Dental Manufacturing Co.
The Woodville Lime Products Co.
Frank W. Yeager

Deaths:

The Society has lost the following 43 members through death (figures in parentheses are dates of membership):

George W. Borton (1909)
C. S. Brainen (1945)
Wheeler P. Davey (1949)
Fred P. Diener (1945)
Murray Erick (1946)
Mariano J. M. Ferraz (1941)
E. B. Fields (1948)
John Fox (1950)
Frederick H. Frankland (1951)
Dan Fretwell (1958)
John R. Geise (1956)
R. H. Harriss (1926)
Raymond H. Hobrock (1936)
Fred A. Jones (1920)
Wilmer H. Koch (1932)
Forrest Ladd (1948)
J. O. Leech (1911)
T. Curtis McKenzie (1958)
P. D. Miesenhelder (1921)
Dalton G. Miller (1924)
W. H. Millspaugh (1945)
Herbert F. Moore (1903)
B. P. Mulcahy (1938)
James A. Murray (1928)
F. L. Parker (1923)
Garnet P. Phillips (1933)
K. T. Potthoff (1917)
Lewis S. Reid (1930)
S. Blake Roberts (1943)
R. W. Roush (1935)
Stephen I. Rudo (1957)
Richard E. Schmidt (1915)
Edward Schramm (1948)
Albert P. Spooner (1920)
Robert A. Stalnaker (1957)
Bradley Stoughton (1902)
Earl C. Sutherland (1940)
Rees F. Tener (1946)
Lambertus van Ouwerkerk (1951)
Robert C. Wheeler (1939)
V. W. Whitmer (1952)
Charles S. Whitney (1929)
F. P. Zimmerli (1929)

In addition to the above, the following representatives of company members of the Society passed away:

R. C. Angell, The S. S. White Dental Manufacturing Co.
M. A. Arnan, The Standards Institution of Israel
Henry C. Ball, Tufted Textile Manufacturers Assn.
R. G. Coleman, Massey-Ferguson, Inc.
M. Dann, Wyeth Laboratories, Inc.
Myrl N. Davis, Kimberly-Clark Corp.
Sherman Doner, Raybestos-Manhattan, Inc.
Martin Frisch, Foster Wheeler Corp.
H. E. Glahn, Dansk Standardiseringsraad
Harry E. Lewis, M. H. Detrick Co.
A. E. Martin, Ainsworth-Precision Castings Co.
C. A. Maynard, The Indiana Steel Products Co.
Bert Meyers, Iowa State Highway Commission
David Nabow, Duke Power Co.
J. E. Orrell, Shell Oil Co.
W. J. Parker, Slide Fastener Assn.
W. E. Robbins, Caribbean Refining Co.
A. B. Smith, Kansas State University
H. C. Stone, Belle City Malleable Iron Co.
William F. Zerbe, Phoenix Steel Corp., Plate Div.

A number of these men had been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. The various issues of the ASTM BULLETIN have included appropriate notices and appreciation of their work. Two of the members whose deaths were reported were practically charter members: Bradley Stoughton, whose membership dates to 1902, and Herbert F. Moore, a past-president and honorary member, who joined the Society in 1903.

Honors and Awards

The Board's Committee on Lectures and Awards has been quite active in reviewing present Society and Committee Awards and in considering policies to be established in connection with future awards. It has recommended a procedure for recognizing outstanding achievements in technical committees, which will be followed as soon as appropriate.

Descriptions of the various existing

awards as well as the personnel of the lecture and award committees are given in the ASTM Year Book.

Recipients of honors and awards to be given at the 1960 Annual Meeting are indicated below:

Honorary Membership:

L. J. Markwardt, retired Assistant Director, U. S. Forest Products Laboratory.

Award of Merit:

Leslie H. Adam
Frank M. Clark
Harold F. Clemmer
O. B. Ellis
Charles M. Gambrill
George R. Gohn
William N. Harrison
Milton E. Holmberg
Lester E. Kuentzel
Stuart M. Phelps
Harry C. Plummer
Orin W. Rees
William A. Reich
Charles H. Scholer
Walter S. Scott
Louis Tanner
Robert R. Thurston
Stanton Umbreit
James S. Vanick

Recognition of Technical Papers:

Charles B. Dudley Medal (research).—G. R. Irvin, J. A. Kies, and H. L. Smith for their paper "Fracture Strengths Relative to Onset and Arrest of Crack Propagation."

Richard L. Templin Award (testing).—J. W. Shupe and W. H. Goetz for their paper "A Laboratory Method for Determining the Skidding Resistance of Bituminous Paving Mixtures."

Sam Tour Award (corrosion).—P. J. Sereda for his paper "Measurements of Surface Moisture."

Sanford E. Thompson Award (concrete, sponsored by Committee C-9).—Stanton Walker and Delmar Bloem for their paper "Variations in Portland Cement."

C. A. Hogentogler Award (soils, sponsored by Committee D-18).—Robert L. Schiffman and Charles R. Wilson for their paper "The Mechanical Behavior of Chemically Treated Granular Soils."

Recognition of Outstanding Contributions:

Frank E. Richart Award (concrete).—Raymond E. Davis, Director Emeritus, Engineering Materials Laboratory, University of California, Berkeley, Calif.

Max Hecht Award (industrial water, sponsored by Committee D-19).—Frank N. Speller, Metallurgical Consultant, Pittsburgh, Pa.

Committee D-12 Award (soaps and other detergents).—Anthony M. Schwartz, Manager, Industrial Chemical Div., Harris Research Laboratories, Washington, D. C.

Harold DeWitt Smith Memorial Award (textiles).—Werner von Bergen, Associate Director of Research, J. P. Stevens & Co., New York, N. Y.

ASTM Lectures:

The *Edgar Marburg Lecture* on "Solar Energy" will be presented on Wednesday afternoon, June 29, at Haddon Hall, Atlantic City, N. J., by Dr. Farrington Daniels, Professor Emeritus, University of Wisconsin, and Vice-President of the National Academy of Sciences.

The *H. W. Gillett Memorial Lecture* on "Nuclear Fuel Elements" will be presented on Tuesday afternoon, June 28, at Haddon Hall, Atlantic City, N. J., by Dr. R. Carson Dalzell, Assistant to the Director of the Division of Reactor Development, United States Atomic Power Commission, Washington, D. C.

Finances

Report for 1959:

Full details of the fiscal operations of the Society for 1959 are included in the report of the auditors appearing in Appendix I.[3]

---

[3] See p. 57.

Operating Receipts:

The budget figure of $1,348,500 was exceeded in actual cash receipts by over $100,000, making a total for the year of $1,448,983. Receipts for dues and entrance fees were $373,583, or 25.8 per cent; sale of publications totaled $964,793 or 66.6 per cent; miscellaneous sources, namely, advertising, income from investments, exhibits, and registration fees, total $110,606 or 7.6 per cent. The increase in the percentage of receipts from sales of publications is largely due to income from the sale of the 1958 Book of Standards.

Operating Disbursements:

Total disbursements were $1,375,051, which included $101,000 held in reserve to complete the payment for the 1959 Supplements to the Book of Standards and the 1959 *Proceedings*, the final bills for which would not be available until 1960.

Total salaries paid were $431,444. While this figure is only 31.3 per cent of the total disbursements, the employee benefits were $62,828. This included health and life insurance, as well as the Society's portion in the Retirement Plan.

Operating Gain:

It was gratifying to have increased the estimated gain from $28,400 to $73,900. This was chiefly because of the increase in sale of publications.

Balance Sheet:

On November 30 the total assets in General Funds were $1,102,822,[4] compared with $996,772 a year ago. The balance sheet takes no account of the inventory of publications in stock. This is consistent with many years' practice. An inventory of the principal technical publications as of May 1, 1960, is summarized below:

Proceedings (all back copies from 1906 to 1958 incl)	2 004
1958 Book of Standards:	
Part 1	5 572
Part 2	6 342
Part 3	7 694
Part 4	7 363
Part 5	6 280
Part 6	3 935
Part 7	4 315
Part 8	4 726
Part 9	6 418
Part 10	5 032
ASTM Methods of Chemical Analysis	558
Special Compilations of Standards	34 755
Selected Standards for Use in College Curricula	5 675
Symposiums, STP's and Special Reprints	258 604
Reprints of Standards (Approx.)	275 000

Building Fund:

Assets in the Building Fund, which include the newly acquired property facing Arch Street, are $446,688.

The General Funds advanced $125,000 for the procurement of the Arch Street property. The total loan advanced to the Building Fund now totals $137,325. It is being amortized at the rate of $17,000 per year as a part of Headquarters Occupancy Cost and appears in the annual operating budget.

Investments:

The operation of the investment fund has continued with no changes. The firm of Loomis, Sayles & Co. is still retained as investment counsel. The income from investments is paid semiannually to the General, Building, Research, etc. Funds in accordance with their pro rata equity.

---

[4] Attention should be called to the fact that whereas the Balance Sheets for both General Funds and Building Fund as shown in the Auditors' Report are correct in themselves, adding the two together in the consolidated statement brings about a duplication of assets with respect to the Cherry Street and Arch Street properties, inasmuch as the money for purchase of the properties was loaned (not given) to the Building Fund and hence still appears as an asset in General Funds in the form of a note, whereas the property purchased with that money also appears as an asset in the Building Fund.

The changes in investments are detailed in the Auditor's Report which is appended.[3] Most of our Mutual Funds have been liquidated, and at this time only 2000 shares of Eaton Howard Balanced Fund are still held. These holdings are being invested largely in bonds.

As of November 30, 1959, the book value or cost of the securities in the fund totaled $907,263. Market value was $1,069,008. Total income was $38,937.

1960 Budget:

The accompanying summary of the 1960 Budget still maintains a conservative estimate for income and a liberal one for costs, so that it is expected the operating balance will be more favorable than that indicated.

BUDGET SUMMARY—1960

		Per cent
Estimated Receipts:		
Dues and Entrance Fees	$388 175	28.9
Publications	810 000	60.3
Miscellaneous	145 500	10.8
TOTAL CURRENT RECEIPTS	1 343 675	100.0
Estimated Disbursements:		
Publications	581 500	41.3
Salaries	485 000	34.3
General Office Expense	121 500	8.6
Technical Committee Expenses	14 500	1.0
Meetings	25 750	1.8
Headquarters Occupancy Expenses	77 500	5.5
Retirement, Insurance Expenses	61 000	4.3
Miscellaneous	45 250	3.2
TOTAL OPERATING DISBURSEMENTS	1 412 000	100.0
ESTIMATED DEFICIT	−68 325	
TOTAL	$1 343 657	

Administrative Matters

A number of matters relating to the administration of the Society are receiving study in the Long-Range Planning Committee. Some of these have to do with Staff organization but they also relate to the scope of activities of the various administrative committees. These activities are now being delineated, including the functioning of the Executive Committee.

Relatively few changes have been made in the Staff personnel aside from the needed additions to the clerical force. The Executive Secretary, R. J. Painter, has been on sick leave in order to recuperate from a hip operation last summer. During this leave the Associate Executive Secretary has been asked to assume the responsibilities of the office.

Headquarters:

Mention was made in the report a year ago of the acquisition of a property on Arch Street adjacent to the present holdings of the Society. This property has been occupied on the instructions of the Board to accommodate a further expansion in the Staff which was not possible in the present Headquarters. An extensive study is being made of Headquarters needs for the immediate future as well as long-range needs so as to permit an early decision concerning the best course to be followed. A subcommittee under the Long-Range Planning Committee, under the chairmanship of E. J. Albert, has been looking into available sites in case it should be decided that a location other than the present one should prove to be more desirable. A complete report on both aspects of the Headquarters expansion can be expected during the coming year.

Group Insurance for Members:

The possibility of making a group health insurance plan available to the members of the Society, similar to that instituted by other professional and technical societies, has been under study for some years. This has culminated in a proposal, administered by Smith, Sternau and Son, that has been brought to the attention of all individual members of the Society. This plan was outlined in the news pages of the ASTM BULLETIN.

Fellowships and Grants-in-Aid:

On the recommendation of the Long-Range Planning Committee, referred to in the report a year ago, a Fellowship and Grants-in-Aid program has been established to further the work of the Society in the field of research and standardization for materials. The University of Illinois was given the first ASTM free-grant doctoral fellowship, which provided for an award of $6500—$1500 for the university and $5000 for the student entering the final year of full-time graduate work on his doctor's degree. Announcement appears in the April issue of the ASTM BULLETIN of the selection of Mr. Roy E. Olson as the recipient of the first fellowship.

In addition to the fellowship, the Society has made five $1000 grants-in-aid, one each to California Institute of Technology, Cornell University, The University of Minnesota, Northwestern University, and The Rice Inst. Descriptions of the projects to which these several grants-in-aid apply will be set forth in the BULLETIN after the schools have made their selections of awardees.

Administrative Committee on Education:

One other action referred to in last year's report resulting from recommendations from the Long-Range Planning Committee was the establishment of an Administrative Committee on Education in Materials. Attention is called to the report of this committee appended to this report.

Division of Materials Sciences

Mention was made in the 1959 report of the Board of the establishment of a new Division of Materials Sciences. This Division is this year sponsoring two extremely interesting symposia which should be of value to a large portion of the Society's membership. As indicated in the various announcements concerning this Division, it is intended that the Division will concern itself with interdisciplinary approaches to the better knowledge of materials, and its activities will relate to subjects that are of interest to a number of the Society's technical committees. It is intended that the work of the new Division will not encroach on the work of the individual technical committees, and to ensure complete coordination in this respect the technical committees are being given an opportunity for representation in the formal organization that is now being effected. A set of by-laws has been drafted to receive the consideration of a meeting to be held during this Annual Meeting, and it is expected that officers of the Division will be elected at that time.

Cooperative Activities

The cooperative activities of the ASTM are so extensive that no attempt will be made to give any enumeration of them. The relations with the American Standards Assn. (ASA) and with the International Organization for Standardization (ISO) are set forth in the appended report of the Administrative Committee on Standards. Much of the effort during the past year has been devoted to keeping up-to-date the approvals as American Standard that have been accorded ASTM standards—seeing that the revisions of standards are approved—nevertheless 16 additional ASTM standards have received approval as American Standard for the first time. It is also of considerable interest that participation in projects of the ISO is being extended. A number of groups have been quite active in effecting the unification of ASTM standards with those of other countries, as set forth in the report of the administrative committee. Recently, on the recommendation of the steel industry, the Board approved for reference to the ASA a recommendation that the United States assume active participation in the international discussions on steel. The Society participates in other international projects as, for example, the International Union of Testing and Research Laboratories for Materials and Construction (RILEM), and it is now engaged in setting up an International Conference on Creep and Fracture.

The ASTM was a participant in the Second National Conference on the Application of Electrical Insulation held in Washington, D. C., under the sponsorship of the American Institute of Electrical Engineers and the National Electrical Manufacturers Assn., and sponsored the program for one of the sessions. Also, the Society sponsored jointly with The American Society of Mechanical Engineers and the American Chemical Society the International Conference on Rubber in Washington, D. C., Nov. 8 to 13, 1959.

The Society participates annually in the Nuclear Congress. This year it sponsored a session at the Congress at which the standardization activities of several organizations were presented, among them ASTM.

The Society is cooperating with the American Society of Agronomy in the preparation of a manual on methods of soil analysis. Similarly, the American Public Health Assn. has requested the cooperation of the Society, along with that of a number of other societies, in preparing a comprehensive laboratory manual in the air pollution field.

There is a mutuality of interest in the work of the ASTM and the Society for Nondestructive Testing, and means for cooperation between the two organizations are being explored.

Very satisfactory working arrangements have been established with the Aerospace Industries Assn. Our Committee E-7 on Nondestructive Testing has the active cooperation of this group in the preparation of some of the radiographic standards that have been published by the Society.

A number of joint committees have been established with other organizations; some of these are of a liaison nature, designed to coordinate the activities of the societies involved, as for example, with the Technical Association of the Pulp and Paper Industry, whereas others are actually working joint committees. A joint committee with the American Society of Civil Engineers on definitions of terms relating to soil mechanics is now being set up.

The Society has many joint activities with the Society of Automotive Engineers.

A recent development of rather especial interest is an arrangement that has been established with *Acta Metallurgica*, an international journal for the science of metals, under which the Society is recognized as a cooperating agency which entitles its members to subscriptions to the journal at considerable discount.

The Board is following with considerable interest the establishment of the societies for testing materials in other countries, such as in Japan and Germany.

Long-Range Planning Committee

Reference has already been made to individual recommendations from the Long-Range Planning Committee and studies that are under way. Apart from these, two subcommittees have been quite active during the year. One, under the chairmanship of R. W. Seniff, has been giving consideration to the organizational set-up of ASTM technical committees and to a review of the Regulations Governing Technical Committees; the other, under the chairmanship of A. T. Goldbeck, is reviewing the present standardization procedure, looking toward its simplification. A third committee is now being appointed to give consideration to the publications set-up of the Society.

R. C. Alden had to relinquish the chairmanship of the Long-Range Planning Committee because of other obligations, and N. L. Mochel has been appointed to serve as chairman.

Society's Name.—The possibility of a change in the name of the Society, mentioned in the report a year ago, was broached in the February issue of the ASTM BULLETIN. In response to a request for comments, a number of letters have been received. Some of these were published in the April BULLETIN, and others will appear in forthcoming issues. It is expected that some decision will be reached within the coming year.

Respectfully submitted on behalf of the Board of Directors,

F. L. LAQUE,
President.

R. E. HESS,
Assoc. Executive Secretary.

June, 1960

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR NOVEMBER 29, 1958 TO NOVEMBER 30, 1959

Philadelphia, January 5, 1960

AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

We have examined the accompanying balance sheet of the American Society for Testing Materials as of November 30, 1959 and the related statements of cash receipts and disbursements of general fund, building fund and investment fund, statement of operations of the general fund, and statements of changes in special and designated funds. Our examination was made in accordance with generally accepted auditing standards, and accordingly included such tests of the accounting records and such other auditing procedures as we considered necessary in the circumstances.

The distribution of income and the determination of participation in the principal of the investment fund were made as of November 16, 1959; therefore, all statements relating to the investment fund and the market value of the securities are reflected as of that date. The securities held in the investment fund and held for the account of other funds were examined by us.

The employees' contributory pension plan established May 1, 1957 is a qualified trust under Section 401(a) of the Internal Revenue Code. During the current fiscal year the Society contributed $45,378 as their share of the cost of the plan. The right is reserved by the Society to amend or discontinue the plan.

It has been the practice of the Society to maintain its accounting records on the general basis of cash receipts and disbursements with the exception that accounts receivable for publication sales and dues receivable from members are recorded, and certain provisions for estimated publication costs and other reserves are also recorded. In addition, the accounts receivable include total billings of orders in those instances in which only partial shipments had been made. This method of keeping the accounts has its limitations in that financial statements prepared from the books do not reflect the Society's financial position at any given date, nor the results of its operations for any definite period as they would be determined from the application of generally accepted accounting principles. Accordingly we are unable to express an over-all opinion on the financial position of the Society at November 30, 1959, or the results of its operations for the period then ended. However, it is our opinion that cash, investments, fixed assets, building fund assets, and special and designated fund assets are presented fairly in the balance sheet, and that the statements of cash receipts and disbursements of, and changes in, the various funds are presented fairly and on a basis consistent with that of the preceding fiscal period.

Respectfully submitted,
Arthur Young & Company

BALANCE SHEET AS OF NOVEMBER 30, 1959

(Including Special and Designated Funds)

ASSETS

General Fund:				
Current:				
Cash			$136 569.41	
Investment Fund Participation (at cost) (market value $785 819.66)			669 649.23	
Accounts Receivable			120 014.16	
Loans Receivable (Building Fund)			137 325.00	
Travel Deposit			425.00	
Total Current Assets—General Fund			1 063 982.80	
Furniture and Fixtures (at Cost Less Accumulated Depreciation)			38 839.86	
Total Assets—General Fund				$1 102 822.66
Building Fund:				
Cash			32 578.77	
Investment Fund Participation (at cost) (market value $37 581.62)			32 025.80	
Land, Cost			138 459.42	
Building and Construction Costs		$342 424.22		
Less, Accumulated Depreciation		98 800.00	243 624.22	
Total Assets—Building Fund				446 688.21
Special and Designated Funds:				
Cash:				
Committee Funds		86 318.95		
ASTM Research Fund		7 077.70		
Dudley Metal and Marburg Lecture Fund	$140.09			
Richard L. Templin Award Fund	287.18			
Sam Tour Award Fund	399.02			
F. E. Richart Award Fund	134.64			
Total Metal and Lecture Funds, Cash		960.93	94 357.58	
Investment Fund Participation (at cost) (market value $270 701.89):				
Committee Funds		129 640.38		
ASTM Research Fund		87 599.32		
Dudley Metal and Marburg Lecture Fund		7 815.94		
Richard L. Templin Award Fund		2 501.11		
Sam Tour Award Fund		1 946.59		
F. E. Richart Award Fund		1 179.75	230 683.09	
Other Investments (at cost):				
Committee Funds (market value $37 612.50)		39 325.00		
ASTM Research Fund (market value $34 667.25)		10 074.63	49 399.63	
Total Assets—Special and Designated Funds				374 440.30
Total Assets				$1 923 951.17

LIABILITIES

General Fund:			
Current:			
Note Payable—Bank		$50 000.00	
Advance Collections		17 586.32	
Due Committees:			
Joint Committee on X-ray Diffraction		258 762.53	
Joint Symposium Nuclear Meeting		1 476.77	
Technical Assistant—C-1		57.41	
Joint Meeting ASTM—Mexico Society		221.35	
Pennsylvania Sales Tax		287.58	
Advance Deposits		490.00	
Provision for:			
1959 Proceedings		40 000.00	
1959 Book of Standards Supplements		81 000.00	
Total Current Liabilities—General Fund		449 881.96	
Reserves:			
Life Membership Fund	$4 339.94		
Publication Fund	16 841.80		
Book of Standards (1961)	100 000.00		
Executive Retirement	13 573.60		
Retirement Fund	36 753.40		
Depreciation of Investments	20 000.00		
Additional Cost of Replacement of Headquarters Building	35 000.00		
Special Annual Meeting Entertainment Account	3 661.64		
Grants-in-Aid	11 500.00		
	241 670.38		
Surplus	411 270.32	652 940.70	
Total Liabilities and Surplus—General Fund			$1 102 822.66
Building Fund:			
Contributions from Members		198 536.49	
Contributions from ASTM General Fund		107 764.57	
Notes Payable—General Fund		137 325.00	
Accumulated Income, Net		3 062.15	
Total Building Fund			446 688.21
Special and Designated Funds:			
Committee Funds, Unexpended Balance		255 284.33	
ASTM Research Fund:			
Principal	100 673.95		
Income	4 077.70	104 751.65	
Dudley Medal and Marburg Lecture Fund:			
Principal	7 815.94		
Income	140.09	7 956.03	
Richard L. Templin Award Fund:			
Principal	2 501.11		
Income	287.18	2 788.29	
Sam Tour Award Fund:			
Principal	1 946.59		
Income	399.02	2 345.61	
F. E. Richart Award Fund:			
Principal	1 179.75		
Income	134.64	1 314.39	
Total Special and Designated Funds			374 440.30
Total Liabilities and Funds			$1 923 951.17

SPECIAL AND DESIGNATED FUNDS

As of November 30, 1959

COMMITTEE FUNDS

Committee A-1	On steel	$2 166.62
Committee A-5	On corrosion of iron and steel	415.98
Committee B-3	On corrosion of non-ferrous metals and alloys	2 471.80
Committee B-4	On metallic materials for electrical heating, electrical resistance, and electrical contacts	96.69
Committee B-5	On copper and copper alloys, cast and wrought	5.04
Committee B-6	On die-cast metals and alloys	3 543.23
Committee B-8	On electrodeposited metallic coatings	15.69
Committee C-1	On cementitious, ceramic, concrete, and masonry materials:	
	General funds	2 049.64
	Cement reference laboratory	(11.54)[a]
	Cement reference laboratory—inspection	2 137.51
Committee C-1–C-9	Cement and concrete reference laboratory	3 000.00
Committee C-9	On concrete and concrete aggregates	170.44
	Sanford E. Thompson Medal Award	1 129.47
Committee C-14	On glass and glass products	51.94
Committee C-15	On manufactured masonry units	591.78
Committee C-16	On thermal insulating materials	212.32
Committee C-17	Flower fund	18.00
Committee C-18	On natural building stones	484.42
Committee D-1	On paint, varnish, lacquer, and related products	836.78
Committee D-2	On petroleum products and lubricants	1 814.96
Committee D-2	ASTM—IBM	1 137.57
Committee D-2	DCC I Reference fuel account	43 469.98
Committee D-3	DCC II National exchange group	(2 337.64)
Committee D-4	On road and paving materials	640.90
Committee D-5	On coal and coke	2 544.77
Committee D-7	On wood	56.47
Committee D-7	Wood pole research fund	1 461.14
Committee D-7	Wood pole—Program #2	224.77
Committee D-9	On electrical insulating materials	2 643.20
Committee D-10	On shipping containers	19.07
Committee D-11	On rubber and rubber-like materials	930.78
Committee D-11	Rubber special	592.46
Committee D-12	On soaps and other detergents	897.11
Committee D-13	On textile materials	2 560.25
Committee D-14	On adhesives	67.54
Committee D-15	On engine antifreezes	23.49
Committee D-18	On soils for engineering purposes	5.62
Committee D-19	On industrial water	196.92
	On research project	6 000.00
Committee D-20	On plastics	851.99
Committee D-27	On electrical insulating liquids and gases	338.83
Committee E-2	On emission spectroscopy	230.80
Committee E-3	On chemical analysis of metals	1.16
Committee E-4	On metallography	42.28
Committee E-7	On nondestructive testing	6 942.22
Committee E-9	On fatigue	315.78
Committee E-14	On mass spectrometry	1 718.85
Committee F-1	On materials for electron tubes and semiconductor devices	203.27
ASA sectional committee on specifications for cast iron pipe and fittings		1 638.19
Chicago district		3 269.76
Cleveland district		229.82
Detroit district		2 467.21
New England district		3 181.30
New York district		6.84
Northern California district		10 339.00
Ohio Valley district		4.85
Philadelphia district		947.47

---

[a] ( ) indicate red figures.

Philadelphia district special (annual meeting account)			3 772.61
Pittsburgh district			2 004.55
St. Louis district			1 020.72
Southern California district			3 360.42
Washington district			218.73
Western New York-Ontario district			1 606.93
Advisory committee on corrosion			42 945.47
Administrative committee on end-use products			322.32
Joint committee on X-ray diffraction:			
Chemical analysis			74 508.81
Research fellowship			2 487.25
Joint committee on leather			2.71
Reserve—future West Coast meetings			7 969.02
			255 284.33
Accounted for as follows:			
Cash in bank and on hand		$86 318.95	
Investments (at cost) (market value $37 612.50):			
$20 000 US Treasury 2½s due 8/15/63	$19 925.00		
20 000 US Treasury 2½s due 11/15/61	19 400.00	39 325.00	
Investment fund participation		129 640.38	$255 284.33

ASTM RESEARCH FUND

Principal Account

	Total	*Invested*	*Uninvested Cash*
Balance, November 28, 1958	$96 229.76	$93 229.76	$3 000.00
Transfer from Income—Stock Dividends	131.43	131.43	
Transfer from Income—Gain on Investments	4 312.76	4 312.76	
Balance, November 30, 1959	100 673.95	97 673.95	3 000.00
Investment Fund Participation	87 599.32		
Other Investments:			
618 shares General Motors Co. (Common) (Note A) (market value $30 977.25)	7 258.63		
48 shares American Telephone & Telegraph (Note A) (market value $3 690.00)	2 816.00	97 673.95	

Income Account

Balance, November 28, 1958	10 220.88	
Receipts:		
Interest, Dividends and Gain on Investments	7 719.47	
Contributions	61.88	18 002.23
Disbursements:		
Transfers to Principal	4 444.19	
Grants:		
Corrosion Research Council	500.00	
ASTM Committee E-7 on Nondestructive Testing	2 200.00	
American Electroplaters Society	500.00	
ASTM Committee D-19 Research Project	6 000.00	
Printing and Stationery	119.87	
Research—University of Delaware	160.47	13 924.53
Balance, November 30, 1959		$4 077.70

Note A.—The income from these securities is paid to annuitants.

DUDLEY MEDAL AND MARBURG LECTURE FUND

Principal Account

Balance, November 28, 1958, Invested		$7 419.40
Transfer from Income—Stock Dividends		11.73
Transfer from Income—Gain on Dividends		384.81
Balance, November 30, 1959, Invested		7 815.94

Income Account

Balance, November 28, 1958, Cash	$193.95	
Receipts:		
Interest, Dividends and Gain on Investments	688.77	
Transfers from General Fund	277.25	1 159.97
Disbursements:		
Transfer to Principal	396.54	
Dudley Medals	108.34	
Marburg Award	500.00	
Marburg Certificates	15.00	1 019.88
Balance, November 30, 1959, Cash		$140.09

RICHARD L. TEMPLIN AWARD FUND

Principal Account

Balance, November 28, 1958, Invested		$2 374.22
Transfer from Income—Stock Dividends		3.75
Transfer from Income—Gain on Investments		123.14
Balance, November 30, 1959, Invested		2 501.11

Income Account

Balance, November 28, 1958, Cash	$256.65	
Receipts:		
Interest, Dividends and Gain on Investments	220.41	477.06
Disbursements:		
Transfer to Principal	126.89	
Awards	50.00	
Certificates	12.99	189.88
Balance, November 30, 1959, Cash		$287.18

SAM TOUR AWARD FUND

Principal Account

Balance, November 28, 1958, Invested	$1 847.84
Transfer from Income—Stock Dividends	2.92
Transfer from Income—Gain on Investments	95.83
Balance, November 30, 1959, Invested	1 946.59

Income Account

Balance, November 28, 1958, Cash	$382.69	
Receipts:		
Interest, Dividends and Gain on Investments	171.54	554.23
Disbursements:		
Transfer to Principal	98.75	
Awards	35.00	
Certificates	21.46	155.21
Balance, November 30, 1959, Cash		$399.02

F. E. RICHART AWARD FUND

Principal Account

Balance, November 28, 1958, Invested	$1 119.90
Transfer from Income—Stock Dividends	1.77
Transfer from Income—Gain on Investments	58.08
Balance, November 30, 1959, Invested	1 179.75

Income Account

Balance, November 28, 1958, Cash	$90.54	
Receipts:		
Interest, Dividends and Gain on Investments	103.95	194.49
Disbursements: Transfer to Principal		59.85
Balance, November 30, 1959, Cash		$134.64

GENERAL FUNDS: CASH RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD NOVEMBER 28, 1958 TO NOVEMBER 30, 1959

Cash Balance, November 28, 1958			$204 595.58
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$360 116.04		
Past Dues	2 219.50		
Advance Dues	3 322.80		
Entrance Fees	7 750.00		
Income, Life Membership	175.00	$373 583.34	
Sales of Publications:			
Book of Standards (Members, Additional Parts)	98 641.21		
Book of Standards, 1955	4 111.65		
Book of Standards, 1958	444 418.68		
Supplement to Book of Standards, 1956–1957	6 351.98		
Supplement to Book of Standards, 1959	7 149.90		
Methods of Chemical Analysis of Metals	5 051.06		
Compilations of Standards	139 414.66		
Separate Standards	51 425.23		
Selected Standards for Students	7 326.64		
Proceedings	8 424.74		
Bulletin Subscriptions (Nonmembers)	6 029.30		
Special and Miscellaneous Publications	186 448.51	964 793.56	
Miscellaneous:			
Advertising—BULLETIN	43 807.46		
Advertising—Index	3 764.04		
Interest and Dividends	29 724.35		
Registration and Other Fees—Meetings	18 490.20		
ASTM Exhibit at Annual Meeting	11 839.75		
Staff Service	2 696.42		
Miscellaneous	909.07	111 231.29	
Nonoperating Receipts (Not Budgeted):			
Excess Remittances	5 347.39		
Committee C-1 for Technical Assistant	3 151.66		
Sale of X-Ray Diffraction Cards	176 861.59		
From Building Fund on Account of Loan	17 000.00		
Gain on Sales of Securities	32 968.69		
Return of Advance Forest Products Laboratory	500.00		
Return of Advance Leeds & Lippincott	2 000.00		
Gillett Memorial	562.27		
Loan from Research Fund	10 000.00		
Loan from Building Fund	10 000.00		
Loan from Girard Trust Corn Exchange Bank	50 000.00		
Annual Meeting—Luncheon	2 333.00		
Annual Meeting—Buffet	339.25		
Miscellaneous	27.58	311 091.43	
Total Receipts			1 760 699.62
Total Receipts for Period November 28, 1958 to November 30, 1959 and Cash Balance November 28, 1958			1 965 295.20

Brought forward			$1 965 295.20

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$4 174.98		
Book of Standards—1955	29.75		
Book of Standards—1958	98 203.61		
Supplement to Book of Standards—1959	3 865.20		
Compilations of Standards	67 449.08		
Methods of Chemical Analyses of Metals	43.07		
Separate Standards	20 280.23		
Proceedings	9 847.02		
Preprints	13 586.94		
ASTM BULLETIN	59 624.76		
Circulars to Members	5 835.33		
Index to Standards	12 636.36		
Special and Miscellaneous and West Coast Publications	135 012.64	$430 588.97	
Salaries	431 444.98		
General Office Expenses	125 383.93		
Expenses—Technical and District Committees	11 797.81		
Expenses—Meetings	41 762.51		
Headquarters Occupancy Expense (Includes $15 000 Depreciation on Buildings)	55 060.00		
American Standards Association	2 250.00		
Traveling Expense, Administrative and Special Committees	15 184.50		
Furniture and Fixtures	5 642.52		
Dues, Contributions, Miscellaneous	1 824.78		
Legal Services, Actuarial and Related Services	5 282.50		
Interest Paid	625.00		
Employees' Retirement Fund	45 877.83		
Group Insurance	4 220.20		
Blue Cross	1 955.63		
Major Medical	2 556.90		
Social Security	8 218.29	759 087.38	
Nonoperating Disbursements (Not Budgeted):			
Refund of Excess Remittances	5 347.39		
Technical Assistant Committee C-1	3 090.42		
Transfer to Joint Committee on X-Ray Diffraction	45 000.00		
Costs and Refunds X-Ray Diffraction Cards	68 562.05		
Executive Retirement Reserve	1 200.00		
Employee Retirement Reserve	290.64		
Book of Standards—1958	79 000.00		
Proceedings—1958	39 000.00		
Investments	33 973.40		
Book of Standards Reserve	161 030.02		
Repayment of Load from Investment Fund	50 000.00		
Repayment of Loan from Research Fund	10 000.00		
Repayment of Loan from Building Fund	10 000.00		
Spring Meeting Luncheon	23.86		
Loan to Building Fund	125 000.00		
Gillett Memorial	566.57		
Annual Meeting—Luncheon	2 922.34		
Annual Meeting—Buffet	473.80		
Special Annual Meeting Account	1 000.00		
Advance for Annual Meeting	2 000.00		
Miscellaneous	568.95	639 049.44	
Total Disbursements			1 828 725.79
Cash Balance, November 30, 1959			$136 569.41

BUILDING FUND: ASSETS AND LIABILITIES

AS OF NOVEMBER 30, 1959

Assets

Cash—Girard Trust Corn Exchange Bank, Philadelphia, Pa.				$32 578.77
Investment Fund Participation (at cost)				32 025.80
Properties (at cost):				
1916–18 Race Street, Philadelphia, Pa.				
Land		$30 000.00		
Building	$26 503.35			
Construction (Net of Salvage)	138 345.83			
Additions 1959:				
New Door in Rear of Building	345.00			
	165 194.18			
Less, Accumulated Depreciation	71 883.05	93 311.13	$123 311.13	
1921–1927 Cherry Street, Philadelphia, Pa. and 1922–1928 Quarry Street, Philadelphia, Pa. (at cost):				
Land		30 800.00		
Building	38 880.10			
Construction	77 463.72			
Additions 1959:				
Opening and Installation of Window	165.00			
	116 508.82			
Less, Accumulated Depreciation	22 116.95	94 391.87	125 191.87	
1921–23 Arch Street, Philadelphia, Pa. and 1920 Cherry Street, Philadelphia, Pa. (at cost):				
Land		70 000.00		
Buildings	59 830.75			
Less, Accumulated Depreciation	4 800.00	55 030.75	125 030.75	
1919 Cherry Street, Philadelphia, Pa. (at cost):				
Land		7 659.42		
Improvements		890.47	8 549.89	382 083.64
Total Assets				446 688.21

Liabilities and Funds

Contributions from Members	198 536.49	
Contributions from ASTM General Fund	107 764.57	
Accumulated Income (Net)	3 062.15	
Loan Payable to ASTM General Fund	137 325.00	$46 688.21

BUILDING FUND: CASH RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD NOVEMBER 28, 1958 TO NOVEMBER 30, 1959

Cash Balance, November 28, 1958			$1 202.43
Receipts			
Contributions from Members		$9 623.84	
Interest and Dividends on Investments		1 245.47	
Profit on Sale of Investments		1 576.71	
Rentals of 1920 Cherry Street		199.50	
Insurance Claim 1916 Cherry Street		225.00	
Loans Payable:			
Girard Trust Corn Exchange Bank		75 000.00	
ASTM General Fund		125 000.00	
ASTM Investment Fund		20 000.00	
From ASTM General Fund:			
On Account of Depreciation on Buildings, Elevator and Air Conditioning Installation		15 000.00	
On Account of Amortization of Note		17 000.00	
On Account of Temporary Advance		10 000.00	274 870.52
			276 072.95
Disbursements			
Purchase of 1921–23 Arch Street and 1920 Cherry Street		114 830.75	
Cherry Street Property:			
New Window Installed	$165.00		
Real Estate Taxes 1959	2 771.15		
Arch Street Property:			
Liability Insurance	21.92		
Real Estate Taxes 1959	1 510.60		
Race Street Property:			
Installation of Door	345.00		
Repayment of Loan from Girard Trust Corn Exchange Bank	75 000.00		
Repayment of Loan from ASTM Investment Fund	20 000.00		
Reduction of Loan from ASTM General Fund	17 000.00		
Temporary Advance to ASTM General Fund	10 000.00		
Transferred to Investment Fund	1 624.76		
Interest on Bank Loan	225.00	128 663.43	243 494.18
Cash Balance, November 30, 1959			$32 578.77

ASTM INVESTMENT FUND

AS OF NOVEMBER 16, 1959

Cash	$25 094.81	
Investments (at cost) (Market value $1 069 008.36)	907 263.31	
Total Assets		$932 358.12
Participations by Funds		$932 358.12

PARTICIPATION BY FUNDS

	Participation November 17, 1958	*Income Reinvested*	*Net Profit, Sale of Securities Reinvested*	*Total Participation November 16, 1959 (at Cost)*
General Fund	$635 675.83	$1 004.71	$32 968.69	$669 649.23
Committee Funds	123 063.32	194.51	6 382.55	129 640.38
Research Fund	83 155.13	131.43	4 312.76	87 599.32
Building Fund	30 401.04	48.05	1 576.71	32 025.80
Medal and Lecture Fund	7 419.40	11.73	384.81	7 815.94
Templin Award Fund	2 374.22	3.75	123.14	2 501.11
Sam Tour Fund	1 847.84	2.92	95.83	1 946.59
Richart Fund	1 119.90	1.77	58.08	1 179.75
	$885 056.68	$1 398.87	$45 902.57	$932 358.12

ASTM INVESTMENT FUND: CASH RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD NOVEMBER 17, 1958 TO NOVEMBER 16, 1959

Cash Balance, November 17, 1958				$33 036.36
Receipts				
Interest and Dividends on Investment		$38 937.29		
Interest on Savings Accounts		71.88		
Total Interest and Dividends			$39 009.17	
Proceeds from Sales of Investments		219 065.30		
Reinvestment of Capital Gain by Participants		45 902.57		
Reinvestment of Stock Dividends by Participants		1 398.87		
Repayment of Loan to Building Fund		20 000.00		
Repayment of Loan to General Fund		50 000.00	336 366.74	
Total Receipts				375 375.91
				408 412.27
Disbursements				
Purchase of Securities			278 405.72	
Investment Counsel			2 750.00	
Loan to Building Fund			20 000.00	
Distribution of Interest and Dividends and Gain to Participants:				
	Interest and Dividends	*Gain*		
General Fund	$26 042.49	32 968.69		
Committee Funds	5 041.69	6 382.55		
Research Fund	3 406.71	4 312.76		
Building Fund	1 245.47	1 576.71		
Medal and Lecture Fund	303.96	384.81		
Templin Award Fund	97.27	123.14		
Sam Tour Fund	75.71	95.83		
Richart Fund	45.87	58.08		
	36 259.17	45 902.57	82 161.74	
Total Disbursements				383 317.46
Cash Balance, November 16, 1959				25 094.81
Balance Composed of:				
Demand Account			22 144.21	
Savings Account			2 950.60	$25 094.81

LIST OF SECURITIES—INVESTMENT FUND

AS OF NOVEMBER 16, 1959

Investments

Number of Shares or Par Value	*Bonds*	*Cost or Book Value*	*Market Value*
$10 000	Commercial Credit Co. 4½ per cent 3/1/77	$9 806.00	$9 000.00
8 000	Consumers Power Co. 1st 4¾ per cent 10/1/87	8 100.00	8 010.00
20 000	General Motors Acceptance Corp. 3½ per cent Deb. 3/15/72	18 250.00	17 550.00
15 000	General Motors Corp. 3¼ per cent Deb. 1/1/79	14 925.00	13 050.00
10 000	Radio Corp. of America 3½ per cent Conv. Sub. Deb. 12/1/80	11 075.00	12 950.00
20 000	Scott Paper Co. 3 per cent Conv. Sub. Deb. 3/1/71	20 377.80	20 550.00
15 000	Socony Mobil Co. 2½ per cent Deb. 6/1/76	13 995.95	11 681.25
15 000	Southern Natural Gas Co. 1st Mtg Pipe Line S F 3⅛ per cent 2/1/74	14 734.81	12 000.00
15 000	Southwestern Bell Telephone Co. 4¾ per cent Debs. 10/1/92	15 450.00	14 625.00
15 000	Superior Oil Co. 3¾ per cent Deb. 7/1/81	15 000.00	13 200.00
10 000	Tennessee Gas Transmission Co. 1st Mtg Pipe Line 4¾s 11/1/76	10 065.00	9 400.00
10 000	U. S. Federal Land Banks 4 per cent 9/20/61	10 027.00	9 837.50
10 000	Federal Land Banks 3⅞ per cent 9/15/72	9 806.25	9 050.00
15 000	Twelve Federal Land Banks 4⅛ per cent 2/15/72–67	15 000.00	14 025.00
15 000	Federal National Mortgage Association 4⅛ per cent 11/12/63	15 002.00	14 587.50
15 000	U. S. Treasury 2½s 11/15/61	15 000.00	14 325.00
15 000	U. S. Treasury 2½s 6/15/67–72	14 694.08	12 459.38
15 000	U. S. Treasury 2½s 8/15/63	14 943.75	13 884.38
20 000	U. S. Treasury 2⅝s 2/15/65	18 664.50	18 187.50
15 000	U. S. Treasury 2¾s 9/15/61	14 934.38	14 475.00
25 000	U. S. Treasury 3⅝s 5/15/61	24 906.25	24 734.38
20 000	U. S. Treasury 3s 8/15/66	18 977.00	18 425.00
20 000	U. S. Treasury 4s 8/1/61	20 056.25	19 850.00
25 000	U. S. Treasury 4⅞s 11/15/63	25 000.00	25 007.81
15 000	U. S. Treasury 5s 8/15/65	15 000.00	15 140.63
20 000	U. S. Treasury, Certificate of Indebtedness 4s 5/15/60	20 033.25	
25 000	U. S. Treasury, Certificate of Indebtedness 4s 5/15/60	25 048.88	44 887.50
30 000	U. S. Savings Bonds Series G, 2½s 6/1/61	30 000.00	29 460.00
5 000	U. S. Savings Bonds Series G, 2½s 1/1/62	5 000.00	4 880.00
5 000	U. S. Savings Bonds Series G, 2½s 2/1/62	5 000.00	4 880.00
	Total Bonds	468 873.15	450 112.83

Preferred Stocks

Shares			
30	Endicott Johnson Corp. 4 per cent Cum Pfd	2 937.50	2 400.00
30	General Motors Corp. $5 Cum Pfd	3 755.80	3 097.50
30	Long Island Lighting Co. 4.35 per cent Series E Cum Pfd	3 015.00	2 512.50
30	P. Lorillard Co. 7 per cent Cum Pfd	4 380.00	4 080.00
30	Mississippi Power & Light Co. 4.36 per cent Cum Pfd	3 075.00	2 520.00
100	Philadelphia Electric Co. 4.4 per cent Cum Pfd	10 670.00	9 200.00
47	Standard Slag Co. 2nd Pfd	2 115.00	2 115.00[a]
30	Westinghouse Electric Corp. 3.80 per cent Cum Pfd Series B	3 067.74	2 460.00
	Total Preferred Stocks	33 016.04	28 385.00

---

[a] Market value not available.

Shares	*Common Stocks*		
150	Abbott Laboratories	8 884.37	9 900.00
202	Allegheny Ludlum Steel Corp	8 111.87	10 201.00
100	Aluminum Co. of America	2 061.45	9 325.00
200	Aluminum, Ltd	6 922.26	5 875.00
100	Amerada Petroleum Corp	10 399.35	7 137.50
250	American Can Co	10 395.00	10 218.75
400	American Metal Climax, Inc	7 271.86	9 700.00
184	American Stores Co	7 712.41	14 352.00
300	American Telephone & Telegraph Co	17 465.36	23 062.50
160	Bethlehem Steel Corp	2 095.99	8 160.00
136	Chase Manhattan Bank	3 930.06	8 279.00
200	Cleveland Electric Illuminating Co	8 271.00	9 600.00
300	Consolidation Coal Co	12 071.32	12 000.00
100	Crown Zellerbach Corp	2 914.79	5 400.00
450	Denver and Rio Grande Western Railroad Co	7 701.73	7 650.00
174.42	Dow Chemical Co	4 034.38	15 725.25
50	E. I. duPont de Nemours & Co	6 544.18	12 650.00
200	Ford Motor Co	11 300.00	15 100.00
125	General Electric Co	4 311.15	10 281.25
450	General Mills, Inc	10 630.89	14 006.25
225	General Telephone & Electronics Corp	3 312.82	16 706.25
150	Great Northern Paper Co	12 688.34	7 425.00
148	Gulf Oil Corp	3 543.27	15 651.00
238	Houston Lighting & Power Co., Texas	8 061.31	15 946.00
200	Johns-Manville Corp	8 063.78	9 850.00
107	Jones Laughlin Steel Corp	2 670.72	7 476.63
100	Merck & Co., Inc	3 887.50	7 625.00
50	National Lead Co	5 059.25	5 281.25
200	Ohio Oil Co	2 623.35	7 075.00
250	Peoples' Gas, Light & Coke	13 632.36	14 625.00
337	Philadelphia Electric Co	9 327.20	17 018.50
100	Philips' Glocilampenfabrieken	8 847.80	9 075.00
500	Potomac Electric Power Co	13 122.32	13 625.00
200	Republic Steel Corp	8 523.26	13 525.00
100	Sherwin-Williams Co	10 515.69	21 500.00
138	Socony Mobil Oil Co	3 103.03	5 502.75
150	Southern Railway Co	5 998.81	7 350.00
153	Square D Co	2 362.37	5 125.50
200	Standard Oil Co. of Indiana	11 969.64	8 075.00
286	Standard Oil Co. of New Jersey	8 971.97	13 191.75
300	Tennessee Gas Transmission Co	10 483.88	9 900.00
282	Texaco, Inc	14 165.58	21 502.50
60	Union Carbide Corp	4 235.24	8 010.00
240	Westinghouse Electric Corp	15 644.26	23 280.00
220	Wisconsin Electric Power Co	7 368.63	8 112.50
	Total Common Stocks	351 211.80	511 078.13
	Mutual Funds		
3 490	Eaton Howard Balanced Fund	54 162.32	79 432.40
	Total Mutual Funds	54 162.32	79 432.40
	Summary		
	Bonds	468 873.15	450 112.83
	Preferred Stocks	33 016.04	28 385.00
	Common Stocks	351 211.80	511 078.13
	Mutual Funds	54 162.32	79 432.40
		$907 263.31	$1 069 008.36

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The standardization activities of the Society continue at quite a rapid rate. From the figures given below concerning the actions taken by the Society at the Annual Meeting and by the Standards Committee throughout the year, and from the accompanying Fig. 1 it will be noted this applies not only to new standards but particularly to the revision of existing standards and tentatives. The latter is of special significance since it is important that the technical committees keep standards under their jurisdiction under constant review to make sure that the standards keep abreast of industrial practices and needs.

It may well be expected there will be a continuing number of new standards issued in view of the organization of quite a number of new committees during the past few years with additional ones authorized during the current year. Some of the newer activities are referred to under Expansion of Standardization Activities.

The several functions of the Administrative Committee on Standards are discussed under the appropriate subdivision headings.

Actions by Standards Committee on Committee Recommendations:

The review of recommendations from technical committees is, of course, one of the primary functions of the Standards Committee, since in its establishment it was intended to provide a means of having recommendations acted upon in the interval between annual meetings· Many of the annual reports refer to recommendations that have been submitted to the Standards Committee during the year. The actions taken this year by the Standards Committee are summarized below:

New Tentatives	39
New Emergency Standards	1
Revised Tentatives	57
Tentative Revisions of Standards	9
Standards Revised and Reverted to Tentative	11
Tentatives Withdrawn	2
Standards Withdrawn	1

A number of these recommendations were acted upon at a meeting held on October 2, 1959; the remainder were handled by correspondence.

Actions by the Society at 1959 Annual Meeting:

As a matter of record, the actions taken by the Society at the 1959 Annual Meeting with respect to standards and tentatives are tabulated below:

New Standards and Existing Tentatives Adopted as Standard	72
Standards in Which Revisions Were Adopted	104
New Tentatives	89
Standards Revised and Reverted to Tentative	14
Tentative Revisions of Standards	15
Tentatives Revised	131
Standards and Tentatives Withdrawn	16

Promotion of Standardization Work:

The Society promotes articles in the BULLETIN discussing various aspects of the standardization work and also the holding of sessions. Of particular interest in this connection is the Symposium on Standards that was sponsored by the Committee on Arrangements at the Third Pacific Area National Meeting of the Society in San Francisco, Calif., Oct. 11–16, 1959. Beyond this it continues to promote having more adequate consideration in committees on the form of standards. The Recommendations on Form of Methods of Testing, under development in Committee E-1 on Methods of Testing, is now being finalized and should soon be available for use by the technical committees.

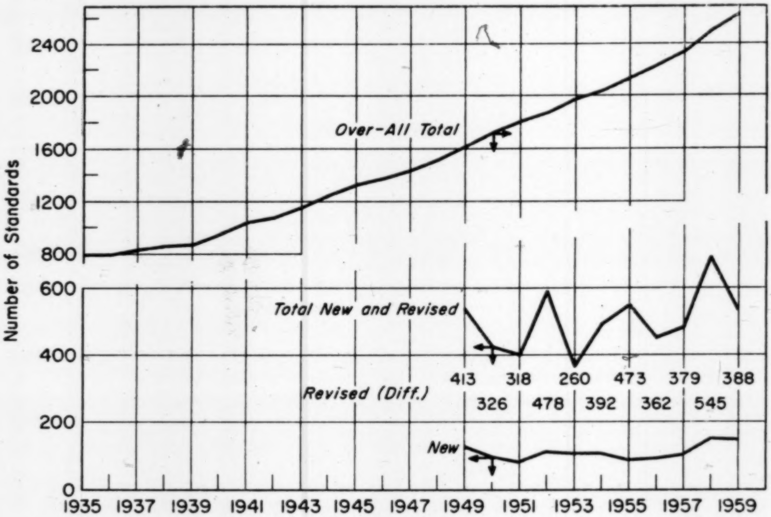


FIG. 1.—Showing Increase in Number of Standards and Tentatives.

EXPANSION OF STANDARDIZATION ACTIVITIES

The Standards Committee has advised the Board of Directors in connection with a number of new standardization projects as set forth below:

Ore Sampling and Analysis.—Reference was made in the report of the committee a year ago of considerations being given to undertaking work in the field of ore sampling and analysis. On the recommendation of the Standards Committee, a new committee was authorized and the organization meeting of Committee E-16 on Sampling and Analysis of Metal-Bearing Ores was held on Jan. 13, 1960, at Society Headquarters.

Graphite.—Mention was made in the previous report of the considerations being given to undertaking work on graphite, specifically the nuclear uses of graphite. Committee C-21 on Ceramic Whitewares and Related Materials has agreed to undertake this work and has organized for the purpose a special task group under Subcommittee V on Nuclear Applications.

Caulking Compounds.—While in the report of the committee a year ago the discussion indicated that a general committee on caulking compounds was contemplated, after reviewing the subject very carefully and on the recommendation of a conference called for the purpose, a new committee was established to deal with the elastomeric type of sealants, leaving to Committee D-1 the work on the oil-base type sealants. The work of the new Committee C-24 on Joint Sealants, however, will be quite broad to include not only the sealants used in building construction, which were the prime considerations when the project was first broached, but also sealants used in aircraft. The assigned scope also includes preformed sealants such as tapes and gaskets.

Gaskets and Packing Materials.—Gaskets as such have received a great amount of consideration during the past two years so as to establish the best procedure to be followed in the Society to cover this important field. While Committee D-11 on Rubber has included gaskets of certain types other than rubber in its deliberations, there had been some thought that the subject was so broad that a separate committee might be required in order that the subject be dealt with from the breadth that would seem to be warranted. Up until the present time, however, there has been no formal recommendation in this connection.

Pigments.—The report a year ago indicated that there may be some need for broadening the work on pigments beyond that being carried out in Committee D-1 on Paint, Varnish, Lacquer and Related Products in view of the use of pigments in other materials such as paper, plastics, and rubber. A conference called to give consideration to such expansion concluded that the study of pigments should not be divorced from their specific application and that the use of pigments in various materials should receive consideration in the individual product committees. In view of Committee D-1's long-standing activity in this field, that committee is being looked to to serve as a coordinating agency for any work on pigments carried out in the other product committees.

Skid Resistance.—At the request of the Highway Research Board, a conference on skid resistance was held at the time of the 1959 Annual Meeting which resulted in an immediate recommendation to the Board of Directors, which was acted upon at its September meeting, authorizing the establishment of a new committee. While skid resistance of highway surfaces is of primary concern, any work in this field would be applicable to airport and walkway surfaces, and the assigned scope of the new Committee E-17 has been worded accordingly.

Ceramics for Electronics.—A field which has come to the fore quite rapidly during the past few years is that of ceramics for electronic uses. A new division on the subject has been established in the American Ceramic Society. In line with the policy of that society to look to the ASTM for standardization work, ASTM was asked to organize a committee to deal with standardization problems relating to the electronic ceramics. On the recommendation of a conference held to give consideration to this request, the Board has authorized a new Committee C-25 on the subject.

Psychophysical Testing.—Much of the discussion in the Administrative Committee on End-Use Products dealt with consumer acceptance. When this administrative committee was discharged, the recommendation was left with the Board that consideration be given to work on psychophysical testing. It developed that a number of the present technical committees of the Society are concerned with various aspects of sensory testing, and a conference called to discuss this subject concluded that a concerted study on some of the basic principles underlying sensory testing would be very worthwhile. It accordingly recommended the organization of a new committee, and Committee E-18 on Sensory Testing has been authorized by the Board. It is now in process of organization.

Hexane of Special Grade.—In view of

the request received from UNICEF in connection with the utilization of proteins such as soybean as a food, Committee D-2 on Petroleum Products and Lubricants has organized a group to insure availability of hexane of a grade of sufficient purity to be used in the processing of such material.

Other Projects.—The Standards Committee still has before it other subjects for new activities in the Society as, for example, methods of gall and seizure testing in which considerable interest has already been expressed; standardization with respect to environmental testing; gas chromatography; radioanalytical procedures.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Association are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship insofar as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, usually with the ASTM as sponsor or co-sponsor of the sectional committee.

Standards Submitted Under the Existing Standards Procedure:

Twenty standards relating to lime were submitted to the American Standards Association for approval as American Standard under the existing standards procedure.

Revised Standards Submitted Under the Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revision of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 6 standards relating to steel, 4 relating to corrosion of iron and steel, 1 relating to cast iron, 1 relating to electrodeposited metallic coatings, 6 relating to manufactured masonry units, 2 to gypsum, 1 to clay pipe, 2 to wood, 1 to rubber, 3 to plastics, and 1 to thermometers.

Standards Submitted Under the Sectional Committee Procedure:

During the year, the following sectional committees, for which the Society is sponsor or co-sponsor, took action to present recommendations to the ASA as follows:

Sectional Committee A1 on Hydraulic Cements.—This committee has recommended the approval as American Standard of 11 ASTM standards, the approval of revisions of 14 American Standards, and the reaffirmation of 1 American Standard.

Sectional Committee A42 on Specifications for Plastering.—This committee has recommended the approval as American Standard of Specifications for Lime-Cement Stucco which have been prepared by the sectional committee.

Sectional Committee B36 on Wrought-Iron and Wrought-Steel Pipe and Tubing.

—This committee has recommended the approval of revisions of 6 American Standards.

Sectional Committee C59 on Electrical Insulating Materials.—This committee has recommended the approval of revisions of 4 American Standards.

Sectional Committee L14 on Textile Test Methods.—This committee has recommended the approval as American Standard of 2 ASTM standards, the approval of revisions of 33 American Standards, and the withdrawal of 1 American Standard.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 1 ASTM standard, the approval of revisions of 11 ASTM standards, and the reaffirmation of 1 American Standard.

INTERNATIONAL RELATIONS

The international discussions, particularly under the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC), are assuming ever greater significance. A number of the European countries apparently wish to have the standardization discussions handled internationally and much of the work is supported here rather than in the development of individual national standards. In certain areas these international discussions are having a definite impact on the standardization in this country and a number of ASTM committees are following these international discussions quite closely with the thought that the ASTM standards and the international recommendations should be in harmony. A list of the various ISO and IEC projects in which ASTM is interested and which continues to grow year by year is given below. Reports appear currently in the ASTM BULLETIN in connection with meetings of these ISO committees, which refer to the actions taken and to the delegations that attend these meetings.

ISO Projects:

Aromatic Hydrocarbons.—ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials is handling participation by the United States in the work of ISO/TC 78 on Aromatic Hydrocarbons for which Great Britain holds the secretariat.

Asbestos-Cement Products.—ASTM Committee C-17 on Asbestos-Cement Products is handling participation by the United States in the work of ISO/TC 77 on Asbestos-Cement Products for which Switzerland holds the secretariat.

Cast Iron.—Great Britain holds the secretariat for ISO/TC 25 on Cast Iron. The Advisory Committee on ASTM Committee A-3 on Cast Iron has been designated to handle American participation.

Chemistry.—Italy holds the secretariat for ISO/TC 47 on Chemistry. Several ASTM committees have indicated an interest in this work, and draft proposals which have been received have been referred to these committees. The coverage of this particular committee is rather broad. The newly organized ASTM Committee E-15 on Industrial Chemicals is interested in a number of aspects of the work of the committee and it now has under consideration recommending that the United States have participating status, at least insofar as test procedures are concerned.

Copper and Copper Alloys.—The United States has found it necessary to relinquish the secretariat for ISO/TC 26 on Copper and Copper Alloys, but it will continue to participate in the work through the Advisory Committee of ASTM Committee B-5 on Copper and

Copper Alloys, which has been designated as the American Group for this ISO work.

Fibre Building Board.—ASTM Committee D-7 on Wood has recommended that United States participation in the work of ISO/TC 89 on Fibre Building Board be limited to that of observer. Germany holds the secretariat for ISO/TC 89.

Fire Tests for Building Materials and Structures.—ASTM Committee E-5 on Fire Tests of Materials and Construction has recommended against United States participation in the work of ISO/TC 92 on Fire Tests for Building Materials and Structures on the basis that such participation could not be justified in view of the expense involved.

Hydraulic Binders.—Sectional Committee A1 on Hydraulic Cements, for which ASTM is sponsor, has recommended participation by the United States in the work of ISO/TC 74 on Hydraulic Binders for which Belgium holds the secretariat. The sectional committee further recommended that ASTM Committee C-1 on Cement handle the United States participation in this work.

Iron and Steel.—ASTM Committee A-1 on Steel has recommended that the United States participate in the work of ISO/TC 17 on Steel and that the group to handle this participation be set up under Committee A-1. Great Britain holds the secretariat for ISO/TC 17.

Light Metals and Alloys.—The Advisory Committee of ASTM Committee B-7 on Light Metals and Alloys has been designated to handle American participation in the work of ISO/TC 79 on Light Metals and Alloys. France holds the secretariat for this ISO committee.

Mica.—Subcommittee IX on Mica of ASTM Committee D-9 on Electrical Insulating Materials has been set up to handle the contacts by the United States with ISO/TC 56 on Mica for which India holds the secretariat.

Paper.—ASTM Committee D-6 on Paper and Paper Products has recommended participation by the United States in the work of ISO/TC 6 on Paper for which France holds the secretariat.

Petroleum Products.—The United States holds the secretariat for ISO/TC 28 on Petroleum Products, the technical work of the secretariat being under the purview of Sectional Committee Z11 on Petroleum Products and Lubricants, for which ASTM is sponsor.

Plastics.—America holds the secretariat for ISO/TC 61 on Plastics. ASTM Committee D-20 on Plastics is handling the work of the secretariat and has organized for the purpose a special committee consisting of representatives of the various groups interested.

Raw Materials for Paints, Varnishes, and Similar Products.—ASTM Committee D-1 on Paint, Varnish, Lacquer and Related Products is handling United States contacts with ISO/TC 35 on Raw Materials for Paints, Varnishes, and Similar Products, for which the Netherlands holds the secretariat.

Refractories.—ASTM Committee C-8 on Refractories is handling United States contacts with ISO/TC 33 on Refractories for which Great Britain holds the secretariat.

Rubber.—The secretariat for ISO/TC 45 on Rubber is held by Great Britain. The American Group for participation in this ISO committee has been set up under ASTM Committee D-11 on Rubber and Rubber-Like Materials.

Shellac.—Subcommittee XIII on Shellac of ASTM Committee D-1 on Paint, Varnish, Lacquer and Related Products has been set up as the American committee in charge of contacts with ISO/TC 50 on Lac for which India holds the secretariat.

Sieves.—Sectional Committee Z23, for which the Society is co-sponsor with the National Bureau of Standards, handles the contacts for the United States in the work of ISO/TC 24 on Sieves.

Solid Mineral Fuels.—ASTM Committee D-5 on Coal and Coke is handling the contacts of the United States with ISO/TC 27 on Solid Mineral Fuels for which Great Britain holds the secretariat.

Surface Active Agents.—ASTM Committee D-12 on Soaps and Other Detergents has recommended participation by the United States in the work of ISO/TC 91 on Surface Active Agents. This has, however, not yet been cleared in the American Standards Association, particularly as to organization of the United States group to handle this participation.

Statistical Treatment of Series of Observations.—The Netherlands holds the secretariat for ISO/TC 69 on Statistical Treatment of Series of Observations. The United States is participating in this work through a special group on which ASTM is represented.

Textiles.—Great Britain holds the general secretariat for ISO/TC 38 on Textiles, but the United States holds the secretariat for several subcommittees. Participation by the United States in this work is being handled by a special committee organized under the sponsorship of ASTM and the American Association of Textile Chemists and Colorists with representation from the various groups interested. This special committee has been given the designation L23.

Viscosity.—The United States holds the secretariat for ISO/TC 66 on Viscosity. The Subcommittee on Rheological Properties of ASTM Committee E-1 on Methods of Testing has been set up to handle the work of the secretariat.

Zinc.—While ASTM Committee B-2 on Non-Ferrous Metals and Alloys had previously recommended active participation by the United States in the work of ISO/TC 18 on Zinc for which Belgium holds the secretariat, it is now recommending participation on the basis of an observer.

IEC Committees:

Insulating Oils.—F. M. Clark, chairman of ASTM Committee D-27 on Electrical Insulating Liquids and Gases, is the technical advisor to the U. S. National Committee of the International Electrotechnical Commission, in connection with the work of IEC/TC 10 on Insulating Oils.

Insulating Materials.—Arnold H. Scott of the National Bureau of Standards is technical advisor to the U. S. National Committee of the IEC for IEC/TC 15 on Insulating Materials. He looks to ASTM Committee D-9 on Electrical Insulating Materials for assistance in processing recommendations from this IEC committee.

Magnetic Steel.—The technical advisor to the U. S. National Committee of the IEC for the work of IEC/TC 14A on Magnetic Steel is A. C. Beiler, chairman of ASTM Committee A-6 on Magnetic Properties.

Aluminum.—E. J. Holcomb of the Aluminum Company of America is technical advisor to the U. S. National Committee for IEC/TC 7 on Aluminum. Mr. Holcomb looks to Sectional Committee C7, for which ASTM is sponsor, for assistance in processing recommendations from this IEC committee.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Administrative Committee on Research held two meetings during 1959: on June 28 in Atlantic City, N. J., and on November 13 at ASTM Headquarters.

W. H. Lutz and R. C. Alden withdrew from the committee during the year, and W. L. Dolch has become a new member.

The research fund is now approximately $100,000. Funds allocated for research projects during the year totaled about $9000. This was largely to assist Committee D-19 on Industrial Water in its project on vaporous carryover of boiler water salts; to assist Committee E-7 on Nondestructive Testing in completion of reference radiographs of cast steel sections; and through Committee D-9 on Electrical Insulating Materials to contribute to a research program sponsored jointly by the National Electrical Manufacturers' Assn., Electronic Industries Assn., and ASTM. Several requests for funds are under consideration. Thus, one from Committee C-4 on Clay Pipe on loading studies of pipe appears to have a rather broad field of application, and cooperation of several other committees may be sought. Reviews were made, also, of various research projects in which ASTM has an interest.

In its consideration of improved means for dissemination of technical information within the Society, the committee has suggested that abstracts of all ASTM technical papers be printed in the ASTM Bulletin in such a way that they can be conveniently clipped and filed, if so desired.

Short articles dealing with some aspect of ASTM research or information dissemination or education were written by members of the committee for the ASTM Bulletin.

A half-day session at the Sixty-second Annual Meeting of the Society in June 1959 was devoted to a forum on research entitled "The Place of Research in ASTM." This covered talks by ACR members on the work and problems of the committee, talks by members of the ASTM Administration on their views of ASTM research, and discussion by many officers and members of the technical committees. It is believed that this has helped bring a better mutual understanding of the problems in fostering and encouraging research in all its ramifications within the Society.

A symposium on "Quality of Observations" is being planned for the Sixty-third Annual Meeting of the Society in collaboration with Committee E-11 on Quality Control of Materials, and is to be issued as a separate technical publication.

Respectfully submitted on behalf of the committee,

Bruce W. Gonser,
Chairman.

Frank Y. Speight,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The Administrative Committee on Papers and Publications held two meetings during the year: on November 19, 1959, and on February 11, 1960.

There have been no changes in the personnel of the committee during the year.

Again the Papers and Publications Committee is reporting a very busy year. While this activity has primarily been due to the very many papers that needed to be reviewed in connection with the Third Pacific Area National Meeting and other meetings, consideration was also given to a number of important general publication matters.

Third Pacific Area National Meeting:

The Third Pacific Area National Meeting was the largest meeting ever sponsored by the Society with 52 sessions and over 230 papers. Not all of these papers are being published, but the program has resulted in the publication of a large number of Special Technical Publications. A number of the subjects were treated as symposia and these lend themselves for publication as Special Technical Publications. Some of the papers are being published in the ASTM BULLETIN.

New Treatment of Special Technical Publications:

Partly as an outgrowth of the West Coast meeting, since several of the symposia were in the same general field, papers in a given field are being grouped so as to have them all appear in a single Special Technical Publication. This is resulting in a number of books of somewhat larger size, but each one will represent more complete coverage. For example, the Symposium on Bituminous Paving Materials (STP 252) comprises the Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders, Symposium on Methods of Test for Design of Bituminous Paving Mixtures, and general papers on Road and Paving Materials, all of which were presented at the 1959 Annual Meeting. A more significant example is the Special Technical Publication on Soils (STP 254) in which papers presented at both the Annual Meeting of the Society and the West Coast meeting are included. Some of the papers presented were in connection with symposia at the Annual Meeting and at the West Coast meeting, whereas others were presented as individual papers in technical sessions at the Annual Meeting. It is felt that handling the papers in this way should result in increasing the stature of the publications and of the individual papers appearing in them.

Book of Standards:

Part 9 of the Book of Standards is increasing so rapidly in size that it will not be feasible to publish in one volume in 1961 all of the standards in the fields covered by this book. Accordingly consideration is being given to splitting this part into two parts: Part 9 and Part 11.

Adjuncts to the Book of Standards:

In view of the great number of comparisons of standards and similar material that do not lend themselves for inclusion in the Book of Standards, a short description of the various material published as auxiliary material has been prepared and is being published in the July issue of the ASTM BULLETIN.

ASTM Bulletin:

The Papers Committee is recommending that the ASTM BULLETIN appear as a regular monthly journal rather than in eight issues as has been the practice in recent years. This should provide the additional coverage that is desired for the increased amount of news material that should be brought to the attention of our members and also a means of publishing currently available papers of general interest.

In the report of the Administrative Committee on Papers a year ago, reference was made to a reader survey then under consideration. A questionnaire was developed and a continuing survey is conducted with each issue of the BULLETIN. A progress report on the survey is given below.

PROGRESS REPORT ON ASTM BULLETIN READERSHIP SURVEY

The original plan for the BULLETIN readership survey was to mail questionnaires to 1200 randomly selected individual members, at the rate of 200 per month. The purpose in asking members' opinion of the BULLETIN was to serve as a guide as to whether this publication is doing the job for which it is intended.

To date, 1000 of the 1200 have been mailed and 516 replies received. A summary of the replies follows:

Content.—The most frequent suggestions were those that asked for more general, "review" articles and those that asked for one or two pages of "briefs" in which the contents of the magazine would be boiled down for the busy reader. A number also asked for more technical papers.

Mechanical Make-up.—Not too many replies fall in this category. Of those that do, the most frequent request is for a new cover design.

Readability.—Not too many replies fall in this category. The most frequent request is for tighter editing, simpler, "less technical" language.

Information in Specific Field.—These replies run nearly the whole gamut of the fields covered by Society activities. Many readers indicated, however, that they realize that the broad range covered by the Society precludes the possibility of covering any one area more thoroughly.

Satisfied.—Nearly 25 per cent said that they were satisfied with things as they are.

1960 Annual Meeting:

A full program is planned for the 1960 Annual Meeting consisting of 37 technical sessions; of these 14 will be devoted to special symposiums as given in the following listing:

Symposium on Recent Progress in Materials Sciences.
Symposium on Nature and Origin of Strength of Materials.
Symposium on Nuclear Methods for Measuring Soil Density and Moisture.
Symposium on Acoustical Fatigue.
Symposium on Shear and Torsion Testing.
Symposium on Methods of Metallographic Specimen Preparation.
Symposium on Radiation Effects and Radiation Dosimetry.
Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials.
Symposium on Quality of Observations.

RECORD OF PUBLICATIONS ISSUED

The extent of the Society's publications during the year is presented in the accompanying Record of Publications.

April 15, 1959 to April 15, 1960

Regular Publications:

1959 Proceedings, 1423 pp., 7500 copies.
1959 Gillett Lecture, 26 pp., 2500 copies.

1959 Marburg Lecture, 32 pp., 3000 copies.

1959 Supplements to Book of Standards:

Part 1, 366 pp., 19,700 copies.

Part 2, 288 pp., 16,150 copies.

Part 3, 116 pp., 15,000 copies.

Part 4, 200 pp., 14,000 copies.

Part 5, 220 pp., 11,600 copies.

Part 6, 144 pp., 8600 copies.

Part 7, 430 pp., 10,150 copies.

Part 8, 254 pp., 9100 copies.

Part 9, 548 pp., 12,000 copies.

Part 10, 396 pp., 9600 copies.

1959 Year Book, 672 pp., 8300 copies.

1959 Index to Standards, 224 pp., 24,000 copies.

ASTM BULLETIN, 8 issues (May 1959 to April 1960), total number of pages 836, average number of copies 17,062 (222 pages of technical papers).

A statement of the volume of the principal publications for the past four years is given below:

	1956–1957, pages	1957–1958, pages	1958–1959, pages	1959–1960, pages
Proceedings	1528	1440	1430	1423
Marburg Lecture	51	40	24	32
Gillett Lecture	32	36	22	26
Book of ASTM Standards	....	....	14 390	....
Supplement to Book of Standards	2335	2644	....	2962
ASTM Methods for Chemical Analysis of Metals	640	....	....	....
Index to ASTM Standards	240	251	240	224
Year Book	640	756	784	672
*Special Technical Publications	2939	3557	3693	3413
*Other Special Publications	108	88	40	12
ASTM BULLETIN	836	840	844	836
Totals	9349	9652	21 443	9600

* Not included under this listing are a number of publications which are partly of the nature of compilations of standards such as:

Building Code Compilation,

Manual of ASTM Standards on Refractory Materials,

Procedures for Testing Soils,

Manual on Industrial Water (STP 148),

Selected ASTM Engineering Materials Standards for Use in College Curricula,

Manual on Measurements and Sampling of Petroleum and Petroleum Products, and Knock Test Manuals.

Special Compilations of Standards Published from April 15, 1959 to April 15, 1960:

Specifications for Steel Piping Materials (A-1), 524 pp., 3000 copies.

ASTM Specifications for Coated and Uncoated Iron and Steel Sheet and Strip (A-1, A-5) 148 pp., 1000 copies.

ASTM Standards on Corrosion of Iron and Steel (A-5), 176 pp., 1000 copies.

ASTM Standards on Metallic Electrical Conductors (B-1), 368 pp., 800 copies.

ASTM Standards on Copper and Copper Alloys (B-5), 720 pp., 1500 copies.

ASTM Standards on Light Metals and Alloys (B-7), 368 pp., 1000 copies.

ASTM Standards on Metal Powders and Metal Powder Products (B-9), 94 pp., 1000 copies.

ASTM Standards on Petroleum Products and Lubricants (D-2), 1232 pp., 7500 copies.

ASTM Standards on Coal and Coke (D-5), 142 pp., 2000 copies.

ASTM Standards on Wood (D-7), 456 pp., 1600 copies.

ASTM Standards on Rubber and Rubber-Like Materials (D-11), 1046 pp., 2500 copies.

ASTM Standards on Electrical Insulating Materials (D-9), 792 pp., 1250 copies.

ASTM Standards on Textile Materials (D-13), 928 pp., 2000 copies.

ASTM Standards on Plastics (D-20), 1244 pp., 3000 copies.

ASTM Methods of Atmospheric Sampling and Analysis (D-22), 112 pp., 1500 copies.

ASTM Standards on Electrical Insulating Liquids and Gases (D-27), 328 pp., 1000 copies.

Manual on Industrial Water and Industrial Waste Water (D-19) (STP 148-D), 670 pp., 2500 copies.

ASTM Manual for Rating Diesel Fuels by the Cetane Method, 136 pp., 1500 copies.

Special Compilations in Prospect:

Building Code Compilation, 270 pp., 1800 copies.

ASTM Manual for Rating Motor Fuels by Motor and Research Methods, 224 pp., 2600 copies.

Special Publications Issued from April 15, 1959 to April 15, 1960:

Index to the X-Ray Powder Data File (STP 48-H), 768 pp., 1000 copies.

X-Ray Powder Data Cards, 800 cards, 700 sets of 3 by 5 index and 500 sets of 4 by 6 Keysort cards (tenth set).

Sixth Supplement to Numerical Index of Abstracted Infrared Spectra, indexed on Wyandotte-ASTM punched cards, 12 pp., 500 copies.

Reference Radiographs for Inspection of Aluminum and Magnesium Castings (Series II), 250 sets of radiographs within a ring binder.

Bibliography of Fatigue References (STP 9-J), 76 pp., 2500 copies.

Index to the Literature on Spectrochemical Analysis—1950–1955, Part IV (STP 41-D), 318 pp., 2500 copies.

Bibliography and Abstracts on Electrical Contacts (STP 56-M), 48 pp., 1500 copies.

Bibliography of Spectrophotometric Methods of Analysis for Inorganic Ions (STP 125-A), 31 pp., 2500 copies.

Symposium on Particle Size Measurement (STP 234), 318 pp., 2000 copies.

Symposium on Paper and Paper Products—New Developments with Accompanying Requirements for New Testing Methods (STP 241) 79 pp., 2000 copies.

Symposium on Bulk Sampling (STP 242), 64 pp., 2000 copies.

Symposium on Materials Research Frontiers (STP 243), 120 pp., 2500 copies.

Symposium on Stability of Distillate Fuel Oils (STP 244), 62 pp., 2000 copies.

Symposium on Plastics Testing and Standardization (STP 247), 279 pp., 3000 copies.

Symposium on Bulk Quantity Measurement (STP 249), 54 pp., 2500 copies.

Symposium on Instrumentation in Atmospheric Analysis (STP 250), 64 pp., 2500 copies.

Symposium on Testing Window Assemblies (STP 251), 52 pp., 2000 copies.

Symposium on Bituminous Paving Materials (includes Symposium on Practical and Statistical Significance of Tests and Properties of Bituminous Binders, Symposium on Methods of Test for Design of Bituminous Paving Mixtures, and general papers on Road and Paving Material) (STP 252), 240 pp., 2000 copies.

Symposium on Identification of Water-Formed Deposits (STP 256), 80 pp., 2500 copies.

Symposium on Microscopy (STP 257), 176 pp., 2500 copies.

Symposium on Visual Aids for Standardizing and Communicating Product Appearance (STP 258), 36 pp., 2000 copies.

Literature Surveys on Influence of Stress Concentrations at Elevated Temperatures and the Effects of Non-Steady Load and Temperature Conditions on the Creep of Metals (STP 260), 110 pp., 2000 copies.

Symposium on Radioisotopes in Metals Analysis and Testing (STP 261), 67 pp., 2500 copies.

Symposium on Electron Metallography (STP 262), 134 pp., 2000 copies.

Symposium on Education in Materials (STP 263), 57 pp., 3000 copies.

Symposium on Electroless Nickel Plating (STP 265), 72 pp., 2000 copies.

Symposium on Hydraulic Fluids (STP 267), 108 pp., 2500 copies.

Special Publications in Prospect:

X-Ray Powder Data cards—Sets 1 to 5 revised, 4000 cards, 500 Keysort and 700 3 by 5 cards.

Index to the X-Ray Powder Data File (STP 48-I), 760 pp., 1000 copies.

Seventh Supplement to the Numerical Index of Abstracted Infrared Spectra, indexed on Wyandotte-ASTM punched cards, 15 pp., 500 copies.

Bibliography and Abstracts on Electrical Contacts (STP 56-N), 50 pp., 1500 copies.

Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (STP 150-C), 30 pp., 1500 copies.

Symposium on High Voltage Cable Insulation (STP 253), 40 pp., 2000 copies.

Soils (includes Symposium on Time Rates of Loading in Soil Testing, Symposium on Atterberg Limits, general papers on Soils, and West Coast Symposium on Soils for Engineering Purposes) (STP 254), 425 pp., 2000 copies.

Marine Atmosphere Exposure Coupled to Form Galvanic Cells Involving Magnesium (STP 255), 46 pp., 1500 copies.

Symposium on Spectroscopic Excitation (STP 259), 48 pp., 2000 copies.

Report on Stress-Corrosion Cracking of Austenitic Steel (STP 264), 80 pp., 2500 copies.

Symposium on Effect of Water-Reducing and Set-Retarding Admixtures on Properties of Concrete (STP 266), 256 pp., 2000 copies.

Symposium on Applied Radiation and Radioisotope Test Methods (STP 268), 150 pp., 2500 copies.

Symposium on Spectroscopy (STP 269), 262 pp., 1500 copies.

Symposium on Durability and Weathering of Structural Sandwich Construction (STP 270), 100 pp., 2000 copies.

Symposium on Adhesion and Adhesives (STP 271), 68 pp., 2000 copies.

Symposium on Newer Metals (STP 272), 266 pp., 2000 copies.

Symposium on Technical Developments in the Handling and Utilization of Water and Industrial Waste Water (STP 273), 120 pp., 2000 copies.

Symposium on Fatigue of Aircraft Structures (STP 274), 120 pp., 2500 copies.

Symposium on Treated Wood for Marine Use (STP 275), 75 pp., 1500 copies.

Symposium on Materials in Nuclear Application (includes papers from Symposium on Ceramics in Nuclear Energy, Symposium on Postirradiation Effects in Polymers, Symposium on Radiation Effects and Dosimetry, and several papers from the Symposium on Technical Developments in the Handling and Utilization of Water and Industrial Waste Water) (STP 276), 300 pp., 3000 copies.

Symposium on Road and Paving Materials (STP 277), 96 pp., 2000 copies.

Symposium on Nondestructive Testing in the Missile Industry (STP 278), 96 pp., 3000 copies.

Symposium on Reinforced Plastics and Thermal Ablation (STP 279), 150 pp., 2000 copies.

Symposium on Bituminous Waterproofing and Roofing Materials (STP 280), 86 pp., 2000 copies.

Symposium on Air Pollution (STP 281), 80 pp., 3000 copies.

Symposium on Building Construction Materials (includes Symposium on Methods for Testing Building Constructions and Symposium on Wood in Building Construction) (STP 282), 100 pp., 2000 copies.

Ten Year Bibliography of Literature on Fatigue, 300 pp., 3000 copies.

Special Joint Publications Issued:

Proceedings of International Rubber Conference—sponsored by ACS, ASME, and ASTM and published by the ACS.

Conference on Explosives—sponsored by the Rocky Mountain District of ASTM and published by the Utah Engineering Experiment Station.

Respectfully submitted on behalf of the committee,

R. E. HESS,
Chairman.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

The Administrative Committee on District Activities held one meeting during the year. At the meeting in June, 1959, E. E. Berkley, A. G. Gray, and L. A. O'Leary were added to the committee. The administrative committee sponsored its annual breakfast on June 24, 1959 for District Council Officers. This proved to be an excellent opportunity for the exchange of information.

The administrative committee conducted a survey for the possible formation of a Northwest District composed of Oregon, Idaho, Washington, and British Columbia. The vote was almost 10 to 1 in favor of such a district, with 43 per cent of the eligible members voting. The committee is studying the formation of such a district and will recommend a provisional council shortly.

The committee plans to study two other problems, namely, revision of the Charter and Manual for District Operation with a view to strengthening the District programs, and a broadening of the scope of District meetings to include national conferences and possibly "workshops."

During the year 15 of the 17 ASTM Districts held meetings for a total of 25. This was well below last year's total of 37. Of the meetings held 16 were jointly sponsored with other technical organizations.

The Student Award Program is now active in 13 districts, one more than last year. The latest district to initiate its program was Central New York. A total of 581 awards were made during the year.

In connection with national meetings, the Chicago District was host to Committee Week held February 1960, during which a dinner and entertainment program was sponsored. The Philadelphia District will be host to the Annual Meeting to be held June 1960 at Atlantic City, N. J. The arrangements will include a Ladies Program and entertainment for the Annual Dinner.

Respectfully submitted on behalf of the committee,

M. N. Clair,
Chairman.

A. L. Batik,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

Two meetings were held during the past year: at Atlantic City, N. J., on June 24, 1959, and at Chicago, Ill., on February 9, 1960. The following new members have been added to the committee: R. H. Carey, Union Carbide Plastics Co.; J. W. Goff, Michigan State University (Packaging); W. J. Hamburger, Fabric Research Laboratories; A. F. Jones, Watertown Arsenal; Douglas McHenry, Portland Cement Assn.; and H. R. Snoke, National Bureau of Standards.

Pursuant to discussions in the committee, a Symposium on Acoustical Fatigue has been developed for the 1960 Annual Meeting. This area is a particularly difficult one in that it involves behavior of material under complex vibration conditions. The subject is of great interest at present in connection with aircraft.

Another subject that has been discussed at length is erosion and cavitation damage of materials. Contacts are being made by various members with respect to specific phases of erosion: automotive valves, rocket nose cones, turbine blades, and concrete. At the next meeting of the committee it will be determined whether to attempt to develop a symposium.

In the packaging field, instances were cited wherein laboratory tests do not correlate satisfactorily with service performance. This will be explored further to determine whether a paper can be prepared which will present the essential facts with respect to status on correlation.

Stress-rupture and embrittling of plastics has been mentioned and will be discussed further.

There are a number of problems associated with performance of building materials, and these will be reviewed, with the possibility of leading to an organized group of papers.

In discussing concrete structures, it was pointed out that the testing of small-scale models has been developed extensively in Europe and is now becoming popular here. The possibility of a joint session with the American Concrete Inst. will be explored.

Several papers submitted to the Society on subjects dealing with simulated service, but borderline to the Society's field, have been reviewed and recommendations made for acceptance or rejection.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

J. W. CAUM,
Secretary.

APPENDIX VII

REPORT OF ADMINISTRATIVE COMMITTEE ON EDUCATION IN MATERIALS

The Administrative Committee on Education in Materials held two meetings during the year—the first on June 23, 1959, at the Annual Meeting in Atlantic City, N. J., and the second on August 5, 1959, at ASTM Headquarters in Philadelphia.

The committee recognizes two types of educational activities in which the Society might be of service to individual members and to the profession. One activity consists in helping to fill the gap between what the engineering graduate has learned in college and what his employer would like him to know about materials when he reports for work. As materials courses become more scientifically oriented, less of the art of using materials will be taught and the gap will widen. Certain industries are now giving on-the-job training to new employees to fill similar gaps in other technical areas. It has been proposed that this committee may be of service in indicating and implementing through ASTM appropriate methods of bridging the gap between theory and practice in the field of materials.

A second activity is directed toward providing the practicing engineer with new and authentic information on modern developments in engineering materials. Often the engineer must solve problems involving new materials or new applications with which he is unfamiliar. Developments in areas such as design of nuclear reactor components, selection of materials for satellites, and solid-state electronic devices require the engineer to become familiar with a wide variety of new materials and applications. Here also, ASTM may perform a valuable service in making available significant information in useful forms.

Several techniques are available for supplying information of the type that will be useful to ASTM members:

1. The Society could sponsor a series of handbooks or monographs for the purpose of (*a*) compiling significant engineering properties of materials—particularly the new materials, or (*b*) describing manufacturing processes and their influence on properties. These publications could be helpful to the neophyte engineer and the practicing engineer as well. In addition, they could serve a useful purpose in design courses in college.

2. The Society could sponsor a series of articles on the science of engineering materials and on engineering applications. Such a series could appropriately be published in the ASTM BULLETIN.

3. Syllabi of courses or programs in the science of engineering materials could be sponsored for use in industry or in guiding college programs. Such syllabi could be particularly helpful in on-the-job training.

4. The Society could sponsor a series of lectures by appropriate individuals. These might be arranged at section or

district meetings. The Finance Committee has ruled that authorization of funds for this purpose would be considered if the district is prepared to support the request.

5. Symposia such as the one on education at the 1959 Annual Meeting and the one to be sponsored by the Materials Sciences Division at the 1960 Annual Meeting should be encouraged.

Coordination should be developed between ASTM activities and the activities of similar groups in other societies and organizations, such as (1) education committees of other professional societies such as ASME, ASCE, AIChE, IAS, ANS, etc., (2) selected industrial organizations such as API, AASHO, NAM, etc., and (3) selected interested companies in specific fields, as aircraft (Boeing, Douglas, Lockheed, MacDonnell, etc.), chemical (DuPont, Goodyear, etc.), civil (State Highway Departments, Army Engineers, etc.), electrical (General Electric, Westinghouse, Bell, RCA, etc.), and nuclear (Argonne, Oak Ridge, Babcock & Wilcox, etc.).

Coordination should be developed between the Administrative Committee on Education in Materials and appropriate and interested technical committees of ASTM. Mr. Bernard Achhammer has been appointed a subcommittee of one to discuss in an exploratory manner with four or five committees the feasibility of having a series of articles written by technical committee members. Mr. F. L. Scoville, Jr., has been appointed by Committee B-2 on Non-Ferrous Metals and Alloys as liaison man with the Administrative Committee on Education in Materials, and Committee A-1 on Steel also plans to appoint a liaison man.

Respectfully submitted on behalf of the committee,

GLENN MURPHY,
Chairman.

FRED F. VAN ATTA,
Secretary.

REPORT OF COMMITTEE A-1
ON
STEEL*

A very significant organizational change was made by Committee A-1 at its January, 1960 meeting. Both the personnel and the activities of the Section on Pipe of Subcommittee XXII were transferred to Subcommittee IX on Steel Tubing and Pipe. This places all activities on steel tubular products in one subcommittee in Committee A-1. Arguments for and against this move were debated several years before final action was taken.

Also at the January, 1960, meeting a task group was appointed by the Advisory Committee to consider active participation by the United States in Technical Committee 17 on Steel of the International Organization for Standardization. For some years the United States has been an observer member of the international group without voting rights. A report of the task group will be made at the June, 1960 meeting of the Advisory Committee, for action by Committee A-1.

Many of the specifications for steel products promulgated by Committee A-1 are adopted by the ASME Boiler and Pressure Vessel Committee for publication in Section II of the Boiler and Pressure Vessel Code. Also many specifications are referenced in case interpretations of the Code. It is very important, therefore, that the specifications reflect the experience of the Code Committee in applying the specification requirements to Code requirements for construction. Many of the proposed revisions in this report, including the marking requirements for exceptions in steel tubular products, fall in this category. The proposed Tentative Specification for Carbon Steel Plates with Improved Transition Properties appended to this report[1] was also developed as the result of a request from the Boiler and Pressure Vessel Committee.

The proposed Tentative Specification for High-Strength Low-Alloy Structural Manganese Vanadium Steel appended to this report[1] was developed in answer to a request from the Bridge Committee of the American Welding Society for a weldable type low-alloy structural steel.

With the design of turbines and generators demanding exacting properties from the rotors, Subcommittee VI on Steel Forgings during the past several years has been developing test methods to measure these properties. The proposed Tentative Method of Test for Notch Toughness of Turbine and Generator Steel Forgings appended to this report[1] is one such test method.

There were many actions on standards taken during the year and referred to the Administrative Committee on Standards. The standards affected are listed later in this report.

Subcommittee XXV on Welding was discharged at the June, 1959, meeting of the Advisory Committee. There have been no problems referred to this group for many years by the committee.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appears in the 1960 Supplement to Book of ASTM Standards, Part 1.

At the January, 1960, meeting of the Advisory Committee, the Special Subcommittee on Hardenability was discharged and its activities transferred to Subcommittee XV on Bar Steels.

Meetings.—Committee A-1 met on June 24, 1959, in Atlantic City, N. J., in conjunction with the 1959 Annual Meeting of the Society. On June 22 and 23, 27 subcommittees, sections, and task groups met to prepare reports for committee action.

Committee A-1 also held a series of meetings in Philadelphia, Pa., on January 18, 19, and 20, 1960. Twenty-six subcommittees, sections, and task forces met on January 18 and 19 with Committee A-1, and the Advisory Committee met on January 20.

Personnel.—The committee consists of 341 voting members, including 150 producers, 143 consumers, and 48 general interest members.

In Subcommittee I on Steel Rails and Accessories, H. H. Morgan, R. W. Hunt Co., and L. H. Winkler, Bethlehem Steel Co., resigned their posts as chairman and vice-chairman respectively after many years of service in these capacities. C. J. Code, The Pennsylvania Railroad Co., and R. B. Stampfle, Bethlehem Steel Co., have been appointed as chairman and vice-chairman.

A. W. MacLaren, U. S. Steel Corp., has resigned as chairman of Subcommittee XV on Bar Steels with H. N. Meyer, Bethlehem Steel Co., named as his successor; also P. A. Archibald, Standard Steel Works Division, Baldwin-Lima-Hamilton Corp., has resigned his post as Chairman of Subcommittee VII on Rolled Steel Wheels and Tires and has been replaced by M. S. Riegel, American Iron and Steel Institute.

In Subcommittee XXII chairman C. L. Kelting, Consolidated Edison Co. of N. Y., vice-chairman W. B. Hoyt, The M. W. Kellogg Co., and secretary C. J. Boyle, General Electric Co., have resigned. Appointed to these posts are chairman J. A. Tash, Duquesne Light Co., vice-chairman H. F. Brown, Esso Research and Engineering Co., and secretary I. A. Rohrig, The Detroit Edison Co.

At the June, 1959, meeting J. G. Morrow, W. S. Scott, and L. H. Winkler were reappointed to two-year terms as members-at-large of the Advisory Committee. Also the chairman of Committee A-10 was appointed a special member of the Advisory Committee in accordance with the recommendation of the Joint Coordinating Committee of Committees A-1, A-9, A-10, B-2, B-4, and F-1.

With much regret Committee A-1 records the death of two honorary members, E. J. Edwards and J. O. Leech. E. J. Edwards was chairman of Subcommittee XI on Steel for Boilers and Pressure Vessels for many years. J. O. Leech was the guiding spirit in developing the basic format of the ASTM steel specifications.

Two other long time members of the Committee who died during the year were F. H. Frankland (Franklin and Lienhard) and A. P. Spooner (Bethlehem Steel Co.). Mr. Frankland's most intense participation was in the field of structural steel; Mr. Spooner's in the work on forgings and castings. Other well-known members whose death the committee records are O. T. Allen (Tennessee Valley Authority), E. B. Fields (Atchison, Topeka, and Santa Fe Railroad System), Chester Hacking (William H. Haskell Mfg. Co.), G. E. Klapper (Philadelphia Electric Co.), and A. D. McLeod (Erie Bolt and Nut Co.).

Election of Officers.—The officers elected for the ensuing term of two years were as follows:

Chairman, J. J. Kanter.

Producer Vice-Chairmen, C. L. Clark and A. O. Schaefer.

Consumer Vice-Chairmen, W. F. Hodges and J. L. Menson.

Secretary, W. Scott.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On September 15, 1959, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations of Committee A-1:

New Tentative Specification for:

Alloy-Steel Turbine Type Bolting Material Specially Heat Treated for High-Temperature Service (A 437 – 59 T).

Revision of Tentative Specifications for:

Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 58 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 58 T),
Carbon and Alloy Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 59 T),
Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 58 T),
Alloy Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 58 T),
Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 58 T),
Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293 – 58 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 58 T),
Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 58 T),
Seamless Ferritic Alloy Steel Pipe for High-Temperature Service (A 335 – 55 T),
Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 58 T),
Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 58 T),
Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 58 T),
Seamless Austenitic Steel Pipe for High-Temperature Central-Station Service (A 376 – 56 T),
Alloy Steel Castings Specially Heat Treated for Pressure Containing Parts Suitable for High-Temperature Service (A 389 – 57 T),
Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 58a T),
Factory-Made Wrought Carbon Steel and Alloy Steel Welding Fittings of Seamless or Welded Construction for Low Temperature Service (A 420 – 58 T), and
Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 – 58 T).

Withdrawal of Standard Specifications for:

Heat-Treated Carbon-Steel Bolting Material (A 261 – 56).

On November 11, 1959, the Administrative Committee accepted the new Tentative Specification for High-Strength Structural Steel (A 440 – 59 T).

The new and revised specifications appear in the 1959 Supplement to Book of ASTM Standards, Part 1.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 3 new tentatives and is recommending the revision of 37 tentatives and 15 standards. Four standards are being revised and reverted to tentative status.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Only a cross-section of the many important activities among the 20 subcommittees will be covered in the following paragraphs:

As a result of requests from many consumers of structural steel, including the

---

[2] See p. 93.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

American Institute of Steel Construction, Subcommittee II has been investigating the possibility of raising the minimum yield point requirement for the standard A 7 steel from the present "33,000" to be "36,000" psi. After discussions with groups such as the American Association of State Highway Officials, American Railway Engineering Assoc., and Association of American Railroads, it has been decided to issue a new specification for structural carbon steel with a minimum yield point of 36,000 psi, suitable for welded, riveted, or bolted structures. The requirements of A 7 steel will not be changed.

Subcommittee VI is developing two new test methods for steel forgings, one for magnetic permeability of generator rotor forgings and the other for magnetic particle inspection of crankshaft forgings.

A task group in Subcommittee VIII considering the weld repair of steel castings has reported that proper control of welding procedure is more important than the amount of welding done. As a result Subcommittee VIII is writing a specification for the repair welding of castings. Reference will be made in the repair provisions of the steel casting specifications to this new document which will cover operator and procedure qualifications and inspection requirements.

The subject of an ASTM specification to replace ASME Boiler and Pressure Vessel Code Case No. 1056 has been reopened in Subcommittee XI. The Tentative Specification for High Tensile Strength Carbon-Manganese Steel Plates for Unfired Pressure Vessels (A 397 - 56 T) was unacceptable to the Code Committee and was withdrawn in 1958.

Subcommittee XIII is preparing a special technical publication describing the various procedures used for measuring the tensile properties of wire strand for prestressing concrete. The subcommittee has discovered that it would be impossible at this time to issue an ASTM test method for a standardized procedure.

Demand has been expressed for a specification for steel sheet for pressure vessels with a composition similar to that of Specification A 212 for steel plates. A proposed specification for high tensile strength carbon silicon steel sheets for pressure vessels has been drafted by Subcommittee XIX.

The ASME Boiler and Pressure Vessel Committee has requested ASTM to consider the development of a specification for high-temperature bolting with expansion characteristics similar to austenitic steel. A joint task group of Subcommittee XII of Committee A-10 and the Section on Bolting of Subcommittee XXII of Committee A-1 has drafted such a specification including alloys A 286, 19-9DL, and EME.

This report has been submitted to letter ballot of the committee, which consists of 341 members; 208 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. KANTER,
Chairman.

H. L. FRY,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Structural Steel (A 36 – 60 T).

Revision of Tentative Specifications for:

Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 59 T),
Alloy-Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 59 T),
Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 60 T),
Carbon and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 59 T),
Carbon and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 – 59 T),
Carbon and Alloy-Steel Forgings for Turbine Bucket Wheels (A 294 – 57 T),
Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 60 T),
Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 59 T), and
Alloy-Steel Castings Specially Heat Treated for Pressure Containing Parts Suitable for High-Temperature Service (A 389 – 59 T).

Revision and Reversion to Tentative of Standard Specifications for:

Alloy-Steel Forgings for Non-Magnetic Coil Retaining Rings for Turbine Generators (A 289 – 55).

The new Tentative Specification A 36 – 60 T was accepted by the Standards Committee on July 28, 1960; the other recommendations were accepted on September 29, 1960. The new and revised tentative specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 1.

APPENDIX

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix, recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. The specifications under revision appear in their present form in either the 1958 Book of ASTM Standards, Part 1, or the 1959 Supplement to Book of ASTM Standards, Part 1.

Marking Requirements for Specification Exceptions

The committee recommends revisions in the following specifications for steel tubular products as explained following the listing:

A 53 - 59 T
A 83 - 59 T
A 106 - 58 T
A 135 - 59 T
A 178 - 59 T
A 179 - 58 T
A 192 - 58 T
A 199 - 58 T
A 209 - 58 T
A 210 - 58 T
A 213 - 59 T
A 214 - 59 T
A 226 - 58 T
A 249 - 59 T
A 250 - 58 T
A 268 - 58
A 312 - 59 T
A 333 - 59 T
A 334 - 59 T
A 335 - 59 T
A 369 - 55 T
A 376 - 59 T
A 423 - 58 T
A 430 - 59 T

Section on Marking.—Add a new paragraph to read as follows:

> When it is specified that certain requirements of these specifications are to be completed by the purchaser upon receipt of the material, the manufacturer shall indicate that all requirements of the specifications have not been completed by a letter X, Y, or Z immediately following the specification number. This letter may be removed after completion of all requirements in accordance with these specifications. An explanation of specification requirements to be completed is provided in Section ——.

Section on Inspection.—Add a new paragraph to read as follows:

> *Certification.*—The certificate or report accomplished by the manufacturer shall include a statement of explanation for the letter added to the specification number (see Section ——), when all the requirements of these specifications have not been completed. The purchaser must certify that all requirements of the specifications have been completed before removal of the letter X, Y, or Z.

New Tentatives

The committee recommends for publication as tentative the following method and specifications as appended hereto:[1]

Tentative Method of Test for:

Notch Toughness of Turbine and Generator Steel Forgings.

Tentative Specifications for:

High-Strength Low-Alloy Structural Manganese Vanadium Steel, and
Carbon Steel Plates with Improved Transition Properties.

Revisions of Tentatives

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for General Requirements for Delivery of Rolled Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use (A 6 - 57 T):

---

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 1.

Section 8 (c).—Change to read as follows:

(*c*) ***Structural-Size Sections and Steel Sheet Piling*** may be conditioned by the manufacturer for the removal of injurious surface imperfections or depressions on their surfaces by grinding, or chipping and grinding, provided the area is ground in a workmanlike manner and the depression does not extend below the rolled surface of the shape more than the following:

$\frac{1}{32}$ in. in material less than $\frac{3}{8}$ in. in thickness, or
$\frac{1}{16}$ in. in material $\frac{3}{8}$ in. and over in thickness.

Surface imperfections, which are greater in depth than the limits shown above, may be removed by chipping or grinding and then depositing weld metal, subject to the following limiting conditions.

(*1*) The total area of the chipped or ground surface of any piece prior to welding shall not exceed 2 per cent of the total surface area of that piece.

(*2*) The reduction of thickness of the material resulting from removal of imperfections at any location, prior to welding, shall not exceed 30 per cent of the nominal thickness at the location of the imperfection; nor shall the depth of depression prior to welding exceed $\frac{1}{2}$ in. in any case.

(*3*) The toes of angles, beams, channels and zees and the stems and toes of tees may be conditioned by grinding or chipping and welding. Prior to welding, the depth of depression, measured from the toe inward, shall be limited to the thickness of the material at the base of the depression, with a maximum depth limit of $\frac{1}{2}$ in.

(*4*) The interlock of any sheet piling section may be conditioned by welding and grinding to correct or build up the interlock at any location not to exceed 2 per cent of the total surface area.

(*5*) All welding shall be performed by qualified welders using low hydrogen welding electrodes conforming to the proper series of the EXX15, EXX16 or EXX18 Classifications of the Tentative Specification for Mild Steel Arc-Welding Electrodes (ASTM Designation: A 233), unless otherwise specified in the basic specification. The electrodes shall be protected from moisture pickup during storage and use. The welds shall be sound, the weld metal being thoroughly fused to all surfaces and edges without undercutting or overlap. Weld metal shall project at least $\frac{1}{16}$ in. above the rolled surface after welding, and the projecting metal shall be removed by grinding, or chipping and grinding to make it flush with the rolled surface and produce a workmanlike finish.

An experienced mill inspector shall inspect the work to see that the imperfections have been completely removed and that limitations specified above have not been exceeded. The inspector representing the purchaser shall, at his option, be given full opportunity to make this same inspection.

Tentative Specification for General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy Steel Bars (A 29 – 59 T):

Section 12.—Change Paragraph (*b*) and add new Paragraphs (*c*), (*d*), and (*e*) to read as follows:

(*b*) If the percentage elongation of any tension test specimen is less than that specified and any part of the fracture is more than $\frac{3}{4}$ in. from the center of a 2-in. specimen, or is outside the middle half of the gage length of an 8-in. specimen, as indicated by scribe scratches marked on the specimen before testing, a retest shall be allowed.

(*c*) For as-rolled material, if the results of any one original tensile specimen are within 2000 psi of the required tensile strength, within 1000 psi of the required yield point, or within 2 per cent of the required elongation, retesting shall be permitted. If the original testing required only one test, the retest shall consist of two random tests from the heat or lot involved. If the original testing required two tests of which one failed by the amounts listed above, the retest shall be made on one random test from the heat or lot. If the results on the retest specimen or specimens meet the specified requirements, the heat or test lot will be accepted. If the results of one retest specimen do not meet the specified requirements, the material is subject to rejection.

(*d*) For thermally treated bars, if the results of the mechanical tests do not conform to the requirements specified, two more tests may be selected for each one failing, and each of these retests shall conform to the requirements of the product specification. However, if necessary, the manufacturer may retreat such lot one or more times, and retests shall be selected in the same manner as the original tests. Each such retest shall conform to the requirements specified.

(*e*) If a bend specimen fails, due to conditions of bending more severe than required by the specification, a retest shall be permitted from the heat or test lot involved for which one random specimen for each original specimen showing failure shall be used. If the results on the retest specimen meet the requirements of the specification, the heat or test lot will be accepted.

Tentative Specifications for Welded and Seamless Steel Pipe (A 53 – 59 T):

Section 7.—Change to read as follows:

7. (*a*) The flattening test shall be made on standard weight and extra strong pipe over 2 in. in nominal diameter. It shall not be required for double extra strong pipe.

(*b*) *Seamless Pipe.*—For seamless pipe a section not less than 2½ in. in length shall be flattened cold between parallel plates in two steps. During the first step, which is a test for ductility, no cracks or breaks on the inside or outside or end surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the following formula:

(retain present formula and definitions)

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material that is revealed during the entire flattening test shall be cause for rejection.

(*c*) *Electric-Resistance-Welded Steel Produced in Single Lengths.*—The crop ends cut from each end of each length shall be flattened between parallel plates until opposite walls of the pipe meet. The tests from each end shall be made alternately with the welds at 0 deg and 90 deg (point of maximum bending). No opening in the weld shall take place until the distance between the plates is less than two thirds of the original outside diameter of the pipe, and no cracks or breaks in the metal, elsewhere than in the weld, shall occur until the distance between the plates is less than one third of the original outside diameter of the pipe. Also, evidence of laminations or burnt metal shall not develop during the entire flattening operation. If any crop end fails to conform to these requirements, additional tests shall be made on specimens cut from the same end of the same length of pipe until the requirements are met, except that the finished pipe shall not be shorter than 80 per cent of its length after the initial cropping. Precautions shall be taken that crop ends can be identified with respect to the length of pipe from which they were cut.

(*d*) *Electric-Resistance-Welded Steel Produced in Multiple Lengths and Subsequently Cut into Single Lengths.*—A flattening test shall be made as described in Paragraph (*c*) on test specimens cut from each end of each multiple length with the weld at 90 deg (point of maximum bending). Tests also shall be made on two intermediate rings cut from each multiple length of pipe with the weld at 0 deg. If any of the specimens fail to conform to the requirements specified in Paragraph (*c*) the manufacturer may elect to make retests cut from each end of each individual length as provided in Paragraph (*c*). The retests for each end of each individual length for electric-resistance-welded steel pipe shall be made with the welds alternately at 0 deg and 90 deg.

(*e*) *Lap Welded and Butt Welded Pipe.*—For lap welded and butt welded pipe a specimen not less than 4 in. in length shall be flattened cold between parallel plates in three steps. The weld shall be located 45 deg from the line of direction of force. During the first step, which is a test for quality of the weld, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than three fourths of the original outside diameter for butt welded pipe or two thirds of the original outside diameter for lap welded pipe. As a second step, the flattening shall be continued. During the second step, which is a test for ductility exclusive of the weld, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than 60 per cent of the original outside diameter for butt welded pipe and not less than one third the original outside diameter for lap welded pipe. During the third step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material or of incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*f*) Surface imperfections in the test specimen before flattening, but revealed during the first step of the flattening test, shall be judged in accordance with the finish requirements in Section 16.

(*g*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

Section 10 (*a*) *and* (*b*).—Change to read as follows:

10. (*a*) One of each of the tests specified in Sections 5, 6, and 7, except electric resistance welded pipe in Section 7, shall be made in one length of pipe from each lot of 500 lengths or fraction thereof of each size.

(*b*) For electric resistance welded pipe the flattening test in Section 7 shall be made on both crop ends from each length of pipe except, when produced in multiple lengths, flattening tests are required only on the crop ends from the front and back of each coil.

Tentative Specifications for Seamless Steel Boiler Tubes (A 83 – 59 T):

New Section.—Add a new Section 3 entitled "Manufacture" to read as follows, renumbering the subsequent sections accordingly:

3. (*a*) Tubes shall be made by the seamless process and shall be either hot-finished or cold-drawn, as specified.

(*b*) Cold-drawn tubes shall be annealed "dead soft" after the final cold-draw pass. Hot-finished tubes need not be annealed unless specified by the purchaser.

Table III.—Add a Note 2 above Table III to read "Tubes smaller than $\frac{1}{2}$ in. in diameter are available upon agreement between manufacturer and purchaser."

Section 5.—Renumber as Section 6 and change to read as follows:

(*a*) A section of tube not less than $2\frac{1}{2}$ in. in length shall be flattened cold between parallel plates in two steps. During the first step, which is a test for ductility, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the following formula:

(retain present formula and definitions)

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the tube meet. Evidence of laminated or unsound material that is revealed during the entire flattening test shall be cause for rejection.

(*b*) Surface imperfections in the test specimen before flattening, but revealed during the first step of the flattening test, shall be judged in accordance with the finish requirements in Section 15.

(*c*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

Section 7.—Renumber as Section 8 and add a new second sentence to read "For tubing less than 1 in. in outside diameter, the length of the specimen shall be $2\frac{1}{2}$ times the outside diameter of the tube."

Section 11.—Renumber as Section 12 and revise the last portion of this section to read "...., retests may be made on additional tubes of double the original number from the same lot, each of which shall conform to the requirements specified."

Tentative Specifications for Electric-Resistance-Welded Steel Pipe (A 135 – 59 T):

Section 7.—Change to read as follows:

7. (*a*) A section of pipe not less than 4 in. in length shall be flattened cold between parallel plates in three steps. The weld shall be placed 90 deg from the direction of the applied force. During the first step, which is a test for ductility of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than two thirds of the original outside diameter of the pipe. As a second step, the flattening shall be continued. During the second step, which is a test for ductility exclusive of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than one third of the original outside diameter of the pipe but is not less than five times the wall thickness of the pipe. During the third step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material or of incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*b*) Surface imperfections in the test specimen before flattening, but revealed during the first step of the flattening test, shall be judged in accordance with the finish requirements in Section 15.

(*c*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

Section 10 (d).—Add a new sentence to read "When pipe is produced in multiple lengths, flattening tests are required only on the crop ends from the front and back end of each coil."

Tentative Specifications for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 58 T):

Section 8.—Revise to read as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 16.

Section 9.—Revise the table on minimum expansion of inside diameter to read as follows, retaining the present footnote *a*:

Ratio of Inside Diameter to Outside Diameter[a]	Minimum Expansion of Inside Diameter, per cent
0.9	21
0.8	22
0.7	25
0.6	30
0.5	39
0.4	51
0.3	68

Tentative Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 59 T):

Section 8.—Revise as indicated above for Section 7 in Specifications A 83.

Section 6.—Change to read as follows:

6. (*a*) A section of tube, not less than $2\frac{1}{2}$ in. in length, shall be flattened cold between parallel plates in two steps. The weld shall be placed 90 deg from the direction of the applied force. During the first step, which is a test for ductility of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the following formula:

(retain present formula and definitions)

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the tube meet. Evidence of laminated or unsound material or of incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*b*) Surface imperfections in the test specimen before flattening but revealed during the first step of the flattening test shall be judged in accordance with the finish requirements in Section 19.

(*c*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

Tentative Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 58 T):

Section 1 (*a*).—Change the words "in sizes $\frac{1}{2}$ in. to but not including 2 in. in outside diameter" to read "in sizes $\frac{1}{8}$ in. to but not including 2 in. in outside diameter."

Section 6.—Revise as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 15.

Section 8 (*a*).—In the first sentence change the last portion to read ". . . but a hardness test shall not be . . . ." Add a second sentence to read "For tubes less than $\frac{1}{4}$ in. in inside diameter, it is permissible to perform the hardness test on the surface of the outside diameter."

Tentative Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 58 T):

Section 6.—Revise to read as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 16.

Section 7.—Revise as indicated above for Section 9 in Specifications A 161.

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 58 T):

Table I.—Under grade B 6 add requirements for AISI type 410 steel to be as follows:

	Range	Check Variation, Over or Under
Carbon, per cent	0.15 max	0.01 under
Manganese, max, per cent	1.00	0.03 over
Phosphorus, max, per cent	0.040	0.005 over
Sulfur, max, per cent	0.030	0.005 over
Silicon, per cent	1.00 max	0.05 over
Chromium, per cent	11.50 to 13.50	0.15

Tentative Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 58 T):

Section 1 (*a*).—Revise as indicated above for Section 1 (*a*) in Specifications A 179.

Table II.—Reletter the present footnote *a* as footnote *b* and add a new footnote *a* referenced after the tensile strength requirement to read "Not applicable to tubes having wall thicknesses below 0.015 in. Tensile properties of such thin wall tubes shall be a matter of agreement between manufacturer and purchaser."

Section 8.—Revise as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 17.

Section 10 (*a*).—Add a sentence to read "For tubes less than $\frac{1}{4}$ in. in inside diameter, it is permissible to perform the hardness test on the surface of the outside diameter."

Tentative Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 58 T):

Section 8.—Revise as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 17.

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 58 T):

Section 8.—Revise as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 19.

Section 9.—Revise as indicated above for Section 9 in Specifications A 161.

Tentative Specifications for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 58 T):

Table I.—Reletter footnote *a* as footnote *b* and add a new footnote *a* referenced after the tensile strength requirement to read "Not applicable to tubes less than $\frac{1}{8}$ in. in outside diameter or having wall thickness below 0.015 in., or both. The tensile properties of such small diameter or thin wall tubes shall be a matter of agreement between the manufacturer and the purchaser."

Table III.—Add a new Note 2 to read "Tubes smaller than $\frac{1}{2}$ in. in outside diameter are available upon agreement between the manufacturer and the purchaser."

Section 8.—Revise as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 19.

Section 9.—Revise as indicated above for Section 9 in Specifications A 161.

Section 10 (*a*).—Revise as indicated above for Section 10 (*a*) in Specifications A 199.

Tentative Specifications for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 59 T):

Section 3.—Add a sentence to read "When bright annealing is used, pickling is not necessary for the austenitic grades."

Section 4.—Replace the present Paragraphs (*a*) and (*b*) by a new Paragraph (*a*) to read as follows, relettering subsequent paragraphs accordingly:

> (*a*) All austenitic material shall be furnished in the heat treated condition. The heat treatment procedure, except for "H" grades, shall consist of heating the material to a minimum temperature of 1900 F and quenching in water or rapidly cooling by other means.

Section 9.—Revise to read as indicated above for Section 5 in Specifications A 83. In the new Paragraph (*b*) refer to Section 20.

Table V.—Revise as indicated above for Table III in Specifications A 210.

Section 11 (*a*).—Revise as indicated above for Section 10 (*a*) in Specifications A 199.

Section 21 (*a*).—Add a new sentence to read "For austenitic steels the marking paint or ink shall not contain any harmful metal or metal salts, such as zinc, lead, or copper, which cause corrosive attack on heating."

Tentative Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 59 T):

Section 6.—Revise as indicated above for Section 6 in Specifications A 178. In the new Paragraph (*b*) refer to Section 17.

Tentative Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 58 T):

Section 6.—Revise as indicated above for Section 6 in Specifications A 178. In the new Paragraph (*b*) refer to Section 18.

Tentative Specifications for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 59 T):

Section 4.—Revise as indicated above for Section 5 in Specifications A 213, except delete the word "austenitic."

Section 9.—Revise as indicated above for Section 6 in Specifications A 178. In the new Paragraph (*b*) refer to Section 22.

Table II.—Revise as indicated above for Table I in Specifications A 210.

Table III.—Revise as indicated above for Table III in Specifications A 83.

Section 12 (*a*).—Revise as indicated above for Section 10 (*a*) in Specifications A 199.

Section 23 (*a*).—Add a new sentence to read "The marking paint or ink shall not contain any harmful metal or metal salts, such as zinc, lead, or copper, which cause corrosive attack on heating."

Tentative Specifications for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 58 T):

Section 8.—Revise as indicated above for Section 6 in Specifications A 178.

Tentative Specification for Carbon Steel Bars Subject to Mechanical Property Requirements (A 306 – 57 T):

Section 10.—Delete Paragraphs (*c*) and (*d*).

Tentative Specification for Stress Relief Annealed Cold-Drawn Carbon Steel Bars (A 311 – 57 T):

Section 9.—Change to read as follows:

9. One tension test shall be made on each lot. A lot shall consist of bars of the same size from the same heat which have been annealed in the same stationary furnace charge. For continuous type of treatment, a lot shall consist of 25 tons or less of the same size of each heat, treated in the same cycle.

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 59 T):

Section 5.—Revise as indicated above for Section 5 in Specifications A 213, except delete the word "austenitic."

Section 11.—Change to read as follows:

11. (*a*) The flattening test shall be made on pipe over 2 in. in nominal diameter.

(*b*) *Seamless Pipe.*—For seamless pipe a section not less than $2\frac{1}{2}$ in. in length shall be flattened cold between parallel plates in two steps. During the first step, which is a test for ductility, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the following formula:

(retain present formula and definitions)

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material that is revealed during the entire flattening test shall be cause for rejection.

(*c*) *Welded Pipe.*—For welded pipe a section of pipe not less than 4 in. in length shall be flattened cold between parallel plates in two steps. The weld shall be placed 90 deg from the direction of the applied force. During the first step, which is a test for ductility of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the formula in Paragraph (*b*). During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material or incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*d*) Surface imperfections in the test specimen before flattening, but revealed during the first step of the flattening test, shall be judged in accordance with the finish requirements in Section 20.

(*e*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

Section 21.—Revise as indicated above for Section 23 (*a*) in Specifications A 249.

Tentative Specification for Quenched and Tempered Carbon Steel Bars (A 321 – 59 T):

Section 13.—Change to read as follows:

13. One tension test shall be made from each tempering or stress-relieving charge. If more than one quenching charge is represented, one tension test shall be made from each quenching charge. If more than one heat is represented in a quenching charge, one tension test shall be made from each heat and/size. For continuous type of treatment, one tension test shall be made for each 25 tons of each heat or size.

Section 14.—Delete this section.

Tentative Specifications for Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 59 T):

Section 11.—Change to read as follows:

(*a*) The flattening test shall be made on pipe over 2 in. in nominal diameter.

(*b*) *Seamless Pipe.*—A section of seamless pipe not less than 2½ in. in length shall be flattened cold between parallel plates in two steps. During the first step, which is a test for ductility, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the following formula.

(retain present formula and definitions)

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material that is revealed during the entire flattening test shall be cause for rejection.

(*c*) *Welded Pipe.*—For welded pipe a section of pipe not less than 4 in. in length shall be flattened cold between parallel plates in three steps. The weld shall be placed 90 deg from the direction of the applied force. During the first step, which is a test for ductility of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than two thirds of the original outside diameter of the pipe. As a second step the flattening shall be continued. During the second step, which is a test for ductility of material exclusive of the weld, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than one third of the original outside diameter of the pipe but is not less than five times the thickness of the pipe wall. During the third step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material or of incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*d*) Surface imperfections in the test specimen before flattening, but revealed during the first step of the flattening test, shall be judged in accordance with the finish requirements in Section 24.

(*e*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

Tables I, II, and III.—Change the designation "Grade C" to read "Grade O."

Section 13 (*a*).—In the table change the designation "Grade C" to read "Grade O."

Tentative Specifications for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 – 59 T):

Tables I, II, III, IV and VI.—Change the designation "Grade C" to read "Grade O."

Section 9.—Change to read as follows:

(*a*) *Welded Tubing.*—For welded tubing a section of tube not less than 4 in. in length shall be flattened cold between parallel plates in two steps. The weld shall be placed 90 deg from the direction of the applied force. During the first step, which is a test for ductility of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the following formula:

(retain present formula and definitions)

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the tube meet. Evidence of laminated or unsound material or of incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*b*) *Seamless Tubing.*—A section of seamless tubing not less than 2½ in. in length shall be flattened cold between parallel plates in two steps. During the first step, which is a test for ductility, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the formula given in Paragraph (*a*).

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the tube meet. Evidence of laminated or unsound material that is revealed during the entire flattening test shall be cause for rejection.

(*c*) Surface imperfections in the test specimen before flattening, but revealed during the first step of the flattening test, shall be judged in accordance with the finish requirements in Section 22.

(*d*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

New Section.—Add a new Section 10 titled "Reverse Flattening Test" to read as follows:

10. A section 4 in. in length shall be taken every 1500 ft of finished welded tubing to be split longitudinally 90 deg on each side of the weld and the sample opened and flattened with the weld at the point of maximum bend. There shall be no evidence of cracks or lack of penetration or overlaps resulting from flash removal in the weld.

Section 11 (*a*).—Renumber as Section 12 and add a new sentence at the end of Paragraph (*a*) to read "For tubes less than ¼ in. in inside diameter, it is permissible to perform the hardness test on the surface of the outside diameter of the tube."

Table III.—Revise the flare test requirements for grade O as indicated above for Section 9 in Specifications A 161.

Tentative Specifications for Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 59 T):

Section 1 (*b*).—Revise to read "One grade of ferritic and eight grades of austenitic steel castings are . . . ."

Table I.—Add grade CF3M with chemical requirements as follows:

	Grade CF3M
Carbon, max, per cent	0.03
Manganese, max, per cent	1.50
Silicon, max, per cent	1.50
Sulfur, max, per cent	0.040
Phosphorus, max, per cent	0.040
Chromium, per cent	17.0 to 21.0
Nickel, per cent	9.0 to 13.0
Molybdenum, per cent	2.0 to 3.0

Table II.—Add grade CF3M with tensile requirements as follows:

	Grade CF3M
Tensile strength, min, psi	70 000
Yield point, min, psi	30 000
Elongation in 2 in., min, per cent	30.0

Tentative Specifications for Heavy-Walled Carbon and Low Alloy Steel Castings for Steam Turbines (A 356 – 58 T):

Section 5.—Change the second sentence to read "This analysis shall be made from (1) the test sample taken during the pouring of the heat, or (2) from a casting representing the heat. If drillings are used, they shall be taken not less than ¼ in. beneath the surface."

Section 6.—Change the second sentence to read "If drillings are used, they shall be taken not less than . . . ."

Section 7 (*b*).—Change to read as follows:

(*b*) Tension tests shall be performed in accordance with the Methods and Definitions for Mechanical Testing of Steel Products (ASTM

Designation: A 370). The yield point shall be determined by use of one of the following methods at a rate of separation of the crossheads under load not to exceed $\frac{1}{16}$ in. per min per in. of gage length:

(*1*) Drop of the beam or halt in the gage,
(*2*) Autographic diagram,
(*3*) Dividers, and
(*4*) Total extension under load using an extensometer.

The tensile strength shall be determined at a rate of separation of the crossheads under load not to exceed $\frac{1}{2}$ in. per min per in. of gage length.

Section 9.—Delete, renumbering subsequent sections accordingly.

Tentative Specifications for Electric-Fusion-Welded Austenitic Chromium-Nickel Alloy Steel Pipe for High-Temperature Service (A 358 – 56 T):

Section 6.—Change to read as follows:

6. (*a*) All pipe shall be furnished in the heat treated condition. The heat treatment procedure shall consist of heating the material to a minimum temperature of 1900 F and quenching in water or rapidly cooling by other means.

(*b*) Controlled structural or special service characteristics shall be specified as a guide for the most suitable heat treatment. If the final heat treatment is at a temperature under 1900 F and is so specified on the order, each pipe shall be stenciled with the final heat treatment temperature in degrees Fahrenheit after the suffix "HT." If no final heat treatment is applied and is so specified on the order, each pipe shall be stenciled "HT-O."

Section 19 (*a*).—Add a sentence to read "Additional marking requirements for heat treatment are described in Section 6 (*b*)."

Tentative Specifications for Seamless Austenitic Steel Pipe for High-Temperature Central Station Service (A 376 – 59 T):

Section 5.—Change to read as follows:

5. (*a*) All pipe shall be furnished in the heat treated condition.

(*b*) *Grades TP304, TP316, TP321, TP347, and TP348.*—The normal heat treatment procedure shall consist of heating the material to a minimum temperature of 1900 F and quenching in water or rapidly cooling by other means. Controlled structural or special service characteristics shall be specified as a guide for the most suitable heat treatment. If the final heat treatment is at a temperature under 1900 F and is so specified on the order, each pipe shall be stenciled with the final heat treatment temperature in degrees Fahrenheit after the suffix "HT." If no final heat treatment is applied and is so specified in the order, each pipe shall be stenciled "HT-O."

(*c*) *Grades TP304H, TP316H, TP321H, and TP347H.*—If cold working is involved in processing, the minimum solution treating temperature for grades TP321H and TP347H shall be 2000 F, and for grades TP304H and TP316H, 1800 F. If the material is hot-rolled, the minimum solution treating temperatures for grades TP321H and TP347H shall be 1925 F, and for grades TP304H and TP316H, 1800 F.

Section 21 (*a*).—Add a sentence to read "Additional marking requirements for heat treatment are described in Section 5 (*b*)."

Tentative Specifications for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 59 T):

Table I.—In footnote *e* change the minimum required yield strength for type 304L from the present "28,000" to read "25,000" psi.

Tentative Specifications for Welded Large Outside Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service (A 409 – 58 T):

Supplementary Requirement S 2.—Change Paragraphs (*a*) and (*b*) to read as indicated above for Section 6 in Specifications A 358.

Section 16 (*a*).—Add a sentence as indicated above for Section 19 (*a*) in Specifications A 358.

Tentative Specifications for Seamless and Electric Welded Low Alloy Steel Tubes for Economizers (A 423 – 58 T):

Section 3.—Revise to read "Tubes made by the seamless process may be cold-drawn or hot-finished."

Section 9.—Revise to read as indicated above for Section 9 in Specifications A 334. In the new Paragraph (*b*) refer to Section 20.

Table III.—Add tolerances for hot finished tubes as shown in the accompanying Table I, retaining the present footnotes.

Section 21 (*a*).—After the reference to designation number add "and whether hot finished or cold drawn."

Section 4 (*b*).—Delete the words "also any alloy element when used."

Section 6 (*a*).—In the table delete the column of requirements for alloy steel.

Section 8 (*b*).—Delete this paragraph.

Section 9.—In the table delete the column of requirements for alloy steel. Delete also all requirements for 1¼ in. bolts.

Section 13 (*b*).—Change to read as follows, retaining the present tabular requirements:

(*b*) *Wrench Turn Fit.*—The nut shall have a free fit for at least two threads in starting on the bolt. When engaged for the thickness of the nut plus two threads, the nut shall show the following minimum and maximum resistance, and for the remainder of the screw length shall not exceed the maximum resistance to turning as expressed in pounds pull applied to the end of a 24-in. wrench.

Section 14.—In Paragraph (*a*) delete the words "together with the initial "A" if alloy bolts."

In Paragraph (*b*) delete Item (*2*) and renumber the subsequent items accordingly.

TABLE I.—PERMISSIBLE VARIATIONS IN DIMENSIONS AND WEIGHT.
(Addition to Table III in Specifications A 423 - 58 T)

Size, Outside Diameter, in.	Outside Diameter (Including Out of Roundness), in.[a, b]		Wall Thickness, per cent[a, c]								Weight[d] per Foot, per cent		Cut Length, in.[e]	
			0.095 in. and under		Over 0.095 to 0.150 in., incl.		Over 0.150 to 0.180 in., incl.		Over 0.180 in.					
	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under
4 and under	1/64	1/32	40	0	35	0	33	0	28	0	16	0	3/16	0
Over 4	1/64	3/64	...	...	35	0	33	0	28	0	16	0	3/16	0

Tentative Specification for Quenched and Tempered Alloy Steel Bars, Hot-Rolled or Cold-Finished (A 434 - 59 T):

Section 15.—Delete this section.

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revision of the following standards and their reversion to tentative status:

Standard Specifications for Heat-Treated Carbon and Alloy Steel Track Bolts and Carbon Steel Nuts (A 183 - 55):

Title.—Delete the words "And Alloy."

Section 1.—Delete the words "And Alloy."

Section 3 (*a*).—In the table delete the column of requirements for alloy steel and delete footnote *a*.

Standard Specifications for Carbon Steel Seamless Drum Forgings (A 266 - 57), Standard Specifications for Carbon and Alloy Steel Forgings for Rings for Reduction Gears (A 290 - 55), and

Standard Specifications for Carbon and Alloy Steel Forgings for Pinions for Reduction Gears (A 291 – 55):

These specifications were extensively revised and are appended hereto in their proposed revised forms.[2]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Open-Hearth Steel Girder Rails of Plain, Grooved, and Guard Types (A 2 – 54):

Section 6.—Change the second sentence to read "This analysis shall be made from a test specimen taken during the pouring of the heat. Determinations may be made chemically or spectrographically."

Standard Specifications for Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 27 – 58):

Section 6.—Change the second sentence to read "The chemical analysis shall be made from a test ingot taken during the pouring of the heat. If drillings are used they shall be taken not less than $\frac{1}{4}$ in. beneath the surface."

Section 7 (a).—Revise the second sentence as indicated above for Section 6 in Tentative Specifications A 356.

Section 8 (b).—Revise as indicated above for Section 7(*b*) in Tentative Specifications A 356.

Section 10.—Delete, renumbering the subsequent sections accordingly.

Standard Specification for Multiple-Wear Wrought Steel Wheels (A 57 – 57):

Section 12.—Change to read "Wheels shall be measured and marked by class to one-half tape sizes and shipped in pairs of the same measured tape size."

Section 13 (h).—In the second sentence delete the word "fillet."

Standard Specifications for Soft Steel Track Spikes (A 65 – 54):

Section 6 (b).—Change to read "The head of the full-size finished spikes shall stand being bent backward to the line of the face of the spikes without showing evidence of forging laps on the surface of the bent portion."

Standard Specifications for Austenitic Manganese-Steel Castings (A 128 – 33):

Section 5.—Revise as indicated above for Section 6 in Standard Specifications A 27.

Section 6.—Change the first three sentences to read "An analysis may be made by the purchaser either spectrographically from a casting representing each heat or by chemical means from samples obtained from a casting representing each heat. If chemical means are used, the samples shall be taken not less than $\frac{1}{4}$ in. below the surface in the case of rough castings. In the case of castings finished by grinding or machining and analyzed by chemical means, the samples for analysis may be taken at the finished surface, provided at least $\frac{1}{4}$ in. of metal has been removed from the casting at the point selected for sampling."

Standard Specification for High-Strength Steel Castings for Structural Purposes (A 148 – 58):

Section 6.—Revise as indicated above for Section 6 in Standard Specifications A 27.

---

[2] The revised specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 1.

Section 7.—Revise as indicated for Section 7 (*a*) in Standard Specifications A 27.

Section 8 (*b*).—Revise as indicated above for Section 8 (*b*) in Standard Specifications A 27.

Section 10.—Delete, renumbering subsequent sections accordingly.

Standard Specification for One-Wear Wrought Steel Wheels (A 186 – 58):

Section 8.—Change to read "Wheels shall be measured and marked by class to one one-half tape sizes and shipped in pairs of the same measured tape size."

Section 9.—Add a new Paragraph (*l*) to read as follows, relettering subsequent paragraphs accordingly "(*l*) One wheel with both tape size and thickness of rim less than specified shall be counted as one wheel in each category."

TABLE II.—CHEMICAL REQUIREMENTS.
(Revision of Table I in Specification A 203 – 56)

	Grade B
Carbon, max, per cent:	
Up to 2 in., incl.	0.21
Over 2 to 4 in., incl.	0.24
Over 4 to 6 in., incl.	0.25
Manganese, max, per cent:	
Up to 2 in., incl.	0.70
Over 2 to 4 in., incl.	0.80
Over 4 to 6 in., incl.	0.80
Nickel, per cent:	
Ladle Analysis	2.10 to 2.50
Check Analysis	2.03 to 2.57

Standard Specification for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 56):

Section 1.—Delete all reference to grade C.

Section 6 (*b*).—Delete all reference to grade C.

Table I.—Delete grade C and revise requirements for grade B to read as shown in the accompanying Table II.

Tables II and III.—Delete grade C.

Standard Specification for High Strength Low Alloy Structural Steel (A 242 – 55):

Section 1.—Change to read as follows:

1. This specification covers high strength low alloy structural steel shapes, plates and bars for welded, riveted or bolted construction intended primarily for use as structural members where savings in weight or added durability are important. These steels have enhanced atmospheric corrosion resistance equal to or greater than carbon structural steels with copper. Welding characteristics vary according to the type of steel furnished. This specification is limited to material up to 4 in., incl., in thickness.

Section 4.—Revise Paragraphs (*b*) and (*c*) and add a new Paragraph (*d*) to read as follows:

(*b*) Choice and use of alloying elements, combined with carbon, manganese, and sulfur within the limits prescribed in Paragraph (*a*) to give the mechanical properties prescribed in Section 5, shall be made by the manufacturer and included and reported in the ladle analysis for information purposes only to identify the type of steel applied.

(*c*) If the steel is specified for materially greater atmospheric corrosion resistance than structural carbon steel with copper, the purchaser should so indicate and consult with the manufacturer.

(*d*) If steel is purchased for welding, the suitability of the chemical composition for welding under the given conditions shall be based on evidence acceptable to the purchaser.

Standard Specifications for Welded and Seamless Open-Hearth Iron Pipe (A 253 – 58):

Section 7.—Change to read as follows:

(*a*) The flattening test shall be made on standard weight and extra strong pipe over 2 in. in nominal diameter. It shall not be required for double extra strong pipe.

(*b*) *Seamless Pipe.*—For seamless pipe a section not less than 2½ in. in length shall be flattened cold between parallel plates in two steps. During the first step, which is a test for ductility, no cracks or breaks on the inside or outside or end surfaces shall occur until the distance between the plates is less than the value of "H" calculated by the following formula:

(retain present formula and definitions)

During the second step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material that is revealed during the entire flattening test shall be cause for rejection.

(*c*) *Electric Resistance Welded Pipe.*—For electric resistance welded pipe a specimen at least 4 in. in length shall be flattened cold between parallel plates in three steps with the weld located 90 deg from the line of direction of force. During the first step, which is a test for ductility of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than two thirds of the original outside diameter of the pipe. As a second step the flattening shall be continued. During the second step, which is a test for ductility exclusive of the weld, no cracks or breaks on the inside or outside surfaces shall occur until the distance between the plates is less than one-third the original outside diameter but not less than five times the wall thickness. During the third step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material or of incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*d*) *Butt Welded Pipe.*—For butt welded pipe a specimen not less than 4 in. in length shall be flattened cold between parallel plates in three steps. The weld shall be located 45 deg from the line of direction of force. During the first step, which is a test for ductility of the weld, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than three fourths of the original outside diameter. As a second step, the flattening shall be continued. During the second step, which is a test for ductility exclusive of the weld, no cracks or breaks on the inside, outside or end surfaces shall occur until the distance between the plates is less than 60 per cent of the original outside diameter. During the third step, which is a test for soundness, the flattening shall be continued until the specimen breaks or the opposite walls of the pipe meet. Evidence of laminated or unsound material or of incomplete weld that is revealed during the entire flattening test shall be cause for rejection.

(*e*) Surface imperfections in the test specimen before flattening, but revealed during the first step of the flattening test, shall be judged in accordance with the finish requirements in Section 16.

(*f*) Superficial ruptures as a result of surface imperfections shall not be cause for rejection.

Standard Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 58):

Section 10 (*a*).—Add a sentence to read "For tube less than $\frac{1}{4}$ in. in outside diameter, it is permissible to perform the hardness test on the surface of the outside diameter of the tube."

Table II.—Add a new footnote *c* referenced after the table title to read:

[c] Not applicable to tubes less than $\frac{1}{8}$ in. in outside diameter or less than 0.015 in. in wall thickness, or both. The tensile properties of such small diameter or thin wall tubes are a matter of agreement between manufacturer and purchaser. For tubing having an outside diameter of $\frac{3}{8}$ in. or less, the gage length shall be four times the outside diameter in order to obtain elongation values comparable to the larger sizes (see the Methods and Definitions for Mechanical Testing of Steel Products (ASTM Designation: A 370)).

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 59):

Section 4.—Change to read as follows:

4. (*a*) All material shall be furnished in the heat-treated condition. The heat treatment procedure shall consist of heating the material to a minimum temperature of 1900 F and quenching in water or rapidly cooling by other means.

(*b*) Controlled structural or special service characteristics shall be specified as a guide for the most suitable heat treatment. If the final heat treatment is at a temperature under 1900 F and is so specified on the order, each tube shall be stenciled with the final heat treatment temperature in degrees Fahrenheit after the suffix "HT." If no final heat treatment is applied and is so specified on the order, each tube shall be stenciled "HT-O."

Section 9 (*b*).—Add a sentence to read "When the Rockwell superficial hardness test is used, as described in Section 11 (*b*), the Rockwell hardness number shall not exceed 76 on the 30T scale or 90 on the 15T scale."

Section 11 (*b*).—Add the following: "For tubing less than 0.065 in. in wall thickness it is permissible to use the Rockwell superficial hardness test. For tubing less than ½ in. in inside diameter, it is permissible to perform the hardness test on the surface of the outside diameter."

Section 17.—Add the following: "Additional marking requirements for heat treating are described in Section 4 (*b*). The marking paint or ink shall not contain any harmful metal or metal salts, such as zinc, lead, or copper, which cause corrosive attack on heating."

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 58):

Section 4.—Change to read as follows:

> 4. All material shall be furnished in the heat-treated condition. The heat treatment procedure shall consist of heating the material to a minimum temperature of 1900 F and quenching in water or rapid cooling by other means.

Section 13.—Add a sentence to read "The marking paint or ink shall not contain any harmful metal or metal salts, such as zinc, lead, or copper, which cause corrosive attack on heating."

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 59):

Section 1 (*b*).—Delete reference to TP348H and TP348.

Section 4.—Revise as indicated above for Section 5 in Tentative Specifications A 376, except delete reference to grades TP316 and TP348 in new Paragraph (*b*) and delete reference to grade TP316H in new Paragraph (*c*).

Table I.—Delete grades TP348 and TP348H.

Section 9.—Revise to read as indicated above for Section 5 in Tentative Specifications A 83. In new Paragraph (*b*) refer to Section 18.

Section 19.—In Paragraph (*a*) delete the reference in the second sentence to grade TP348H. Also in Paragraph (*a*) add a new sentence to read "Additional marking requirements for heat treating are described in Section 4 (*c*)."

In Paragraph (*b*) replace the words "lead, copper, etc." with "zinc, lead, or copper."

Standard Specifications for Carbon and Alloy Steel Forgings for Pressure Vessel Shells (A 372–58):

Table I.—Add requirements for new type E under Class V as follows:

	Ladle Analysis	Check Analysis
Carbon, per cent	0.35 to 0.50	0.32 to 0.53
Manganese, per cent	0.75 to 1.05	0.70 to 1.10
Phosphorus, max, per cent	0.04	0.05
Sulfur, max, per cent	0.04	0.05
Silicon, per cent	0.15 to 0.35	0.13 to 0.37
Chromium, per cent	0.80 to 1.15	0.74 to 1.21
Molybdenum, per cent	0.15 to 0.25	0.12 to 0.28

REPORT OF COMMITTEE A-3
ON
CAST IRON*

Committee A-3 on Cast Iron held two meetings during the year: in Atlantic City, N. J., on June 24, 1959, and in Chicago, Ill. on Feb. 4, 1960. A number of subcommittees and task groups met in conjunction with the main committee and between meetings of the main committee.

The committee consists of 122 members, of whom 118 are voting members; 44 are classed as producers, 42 as consumers, and 32 as general interest members. There is 1 consulting member.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. A. Clark.
Vice-Chairman, H. W. Lownie, Jr.
Secretary, C. F. Walton.
Membership Secretary, W. M. Spear.

During the year, the committee lost because of death the services of two long-time and valued members: G. P. Phillips and E. B. Fields.

The committee formed the new Task Group on Thermal Fatigue under W. M. Spear, and disbanded its 3-year-old Task Group on Test Bars as having completed its assignment. Subcommittee 26 on Low-Temperature Properties of Cast Iron was disbanded because of insufficient interest.

New subcommittee chairmen appointed during the year included W. S. Mabb (Subcommittee 16 on Heat Treatment of Cast Iron), R. A. Clark (Subcommittee 18 on Automotive Castings), J. T. Vann (Subcommittee 21 on Pressure Pipe) and J. R. Kattus (Subcommittee 22 on Elevated Temperature Properties of Cast Iron). The committee offered special thanks to J. S. Vanick, the outgoing chairman of Subcommittee 22, for long and active service.

Mr. R. A. Clark was appointed as the representative of Committee A-3 on SAE Panel B Division 9 on Automotive Iron Castings, and George Krumlauf was confirmed as the representative of Committee A-3 on ASTM Committee E-3 on Chemical Analysis of Metals.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee A-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method for:

Transverse Testing of Gray Cast Iron (**A 438 – 60 T**).

Revision and Reversion to Tentative of Standard Specifications for:

Gray Iron Castings (**A 48 – 56**).

These recommendations were accepted by the Standards Committee on April 14, 1960, and the new and revised tentatives are available as separate reprints.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

Tentative Specifications for:

Austenitic Nodular Iron Castings, and
Ferritic Nodular Iron Castings for Valves, Flanges, Pipe Fittings, and Other Piping Components.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Foundry Pig Iron (**A 43 – 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Chill Testing of Cast Iron (**A 367 – 58**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Figure 1.—Revise to add between wedges 3 and 4 a new wedge No. W 3½, with the following dimensions: B, 1.0 in.; H, 1.75 in.; A, 32 deg; and L, 5 in.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Task Group on Chemical Analysis of High-Carbon Irons (George Krumlauf, chairman) is investigating ASTM methods of sampling and chemical analyses of high-carbon irons.

Task Group on International Standards (D. E. Krause, chairman) has maintained contact by correspondence with ISO Technical Committee 25, but has continued to be unable to finance representation at meetings of TC25. As a result of study and letter ballots, the Task Group recommended to ASA that affirmative votes (with comments) be cast on ISO Draft Recommendations 220 on Gray Cast Iron and on ISO Draft Recommendations 286 on Brinell Hardness Test of Gray Cast Iron. Currently, the Task Group is studying U. S. practices on impact testing of cast iron.

Task Group on Test Bars (G. A. Timmons and H. W. Lownie, Jr., cochairmen), made up of members of Subcommittee 6 on General Castings and Subcommittee 11 on Methods of Testing, issued a final report recommending (1) that Specifications A 48 – 56 be revised to permit the use of short test bars for tension testing to replace long "transverse" bars, and (2) that transverse testing of gray iron be covered in a separate method of testing. The report was accepted, the task group was disbanded, and the recommendations of the task group were referred to the appropriate subcommittees for action.

Task Group on Thermal Fatigue (W. Spear, chairman) was formed to investigate the need for research and further investigation of thermal fatigue as it applies to cast iron.

Subcommittee 1 on Pig Iron (H. W. Stuart, chairman) recommended the adoption of Specifications A 43 as standard, as noted earlier in this report.

Subcommittee 6 on General Castings (H. W. Lownie, Jr., chairman) completed the major tasks of (1) completely rewriting Specification A 48 to permit the use of short tension-test bars, and (2) originating a new method of transverse testing.

---

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 1.

[2] 1958 Book of ASTM Standards, Part 1.

[3] 1958 Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee 12 on Austenitic Cast Iron (Hugo Larson, chairman) completed the new Tentative Specifications for Austenitic Nodular Iron Castings listed earlier in this report. On the latter activity, Subcommittee 12 acted as a Task Group under Subcommittee 24.

Subcommittee 18 on Automotive Castings (R. A. Clark, chairman) began a review and restudy of Specifications A 159 with the idea of bringing it up to date and of correcting deficiencies noted in two years of use. Subcommittee 18 will work closely with SAE Panel B Division 9 on this revision.

Subcommittee 19 on Chilled and White Iron Castings (N. R. Arant, chairman) has a task group actively at work preparing a specification on chilled iron rolls for the rubber industry.

Subcommittee 20 on Soil Pipe and Fittings (J. W. Perry, chairman) is being reorganized.

Subcommittee 22 on Elevated-Temperature Properties of Cast Iron (J. R. Kattus, chairman) will continue to report to Committee A-3 the activities of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals, particularly on new research involving the elevated-temperature properties of austenitic nodular iron. Subcommittee 22 also is considering the question of what action Committee A-3 should take relative to research on thermal fatigue of cast iron.

Subcommittee 24 on Nodular Iron (H. W. Ruf, chairman) has completed the proposed new tentatives on (1) austenitic nodular iron castings, and (2) ferritic nodular iron castings for valves, flanges, pipe fittings, and other piping components, referred to earlier in the report. Task groups are actively studying (1) possible revision of Specifications A 339, and (2) cast-to-size test bars for nodular iron.

Subcommittee 25 on Methods of Chill Testing Cast Iron (George Krumlauf, chairman) proposed the revision of Methods A 367 to add a needed new size of test specimen, as mentioned earlier in the report.

This report has been submitted to a letter ballot of the committee, which consists of 118 voting members; 87 members returned their ballots, of whom 86 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. E. EAGAN,
Chairman

H. W. LOWNIE, JR.
Secretary.

REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held two meetings during the past year: on June 23, 1959, at Atlantic City, N. J., and on Feb. 3, 1960, at Chicago, Ill.

The committee consists of 109 members, of whom 53 are classified as producers, 30 as consumers, and 26 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. F. Hormann.
First Vice-Chairman, E. K. Camp.
Second Vice-Chairman, J. F. Occasione.
Secretary, C. P. Larrabee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee A-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils, and Cut Lengths (A 93 - 58 T),
Long Terne Iron or Steel Sheets, Coils, and Cut Lengths (A 308 - 58 T), and
1.25 oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 - 59 T).

These recommendations were accepted by the Standards Committee on October 27, 1959, and the revisions appear in the 1959 Supplement to Book of ASTM Standards, Part 1.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Tentative Specification for Zinc-Coated (Galvanized) Iron or Steel Sheets for Culverts and Underdrains as appended hereto.[1]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Specifications for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (**A 153 - 59**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot vote of the Society. The recommended changes are intended to preclude the use by industry of embrittled malleable iron hardware.

Table I.—In the first column delete the words "Gray iron" from the class A Material.

Section 7.—Add a new Paragraph (*b*) to read as follows, relettering subsequent paragraphs accordingly:

> (*b*) Malleable castings shall be of such composition as will preclude the possibility of galvanizing embrittlement, or they shall be either

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 1.

[2] 1959 Supplement to Book of ASTM Standards, Part 1.

cooled from the anneal, or subsequently heat treated so as to immunize them against such embrittlement.

In the present Paragraph (*c*) add the word "steel" in the first sentence to read: "A convenient bend test for embrittlement of galvanized steel hardware such as . . . ."

Change the present Paragraph (*d*) to read as follows:

> (*d*) Castings and steel hardware, of such shape or size that they do not permit bending, may be struck a sharp blow with a hammer of suitable weight. If the material is not embrittled, it should not crack under such a blow. However, if the article is tested to destruction, a white lustrous, coarse grain appearance of the resulting fracture is indicative of embrittlement and shall be cause for rejection.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Methods of Test for Weight of Coating on Aluminum-Coated Iron or Steel Articles (**A 428 – 58 T**).[3]

EDITORIAL CHANGE

The committee recommends the following editorial change in the Tentative Specifications for Zinc-Coated Steel Chain-Link Fabric (**A 392 – 59 T**):[2]

Section 7(b).—Change the first sentence to read: "Specimens for testing shall consist of a 1 ft sq of woven fencing . . . "

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Testing (O. B. Ellis, chairman).—Investigations have been conducted on determination of weight of aluminum coatings on steel by stripping using NaOH-HF, and HCl-SbCl$_3$. The tests disclosed that the sodium hydroxide - hydrofluoric acid method was no better than the NaOH-HCl method now specified in Methods A 428, and as it involves the use of a more hazardous chemical it is not recommended. The hydrochloric acid - antimony chloride method was not satisfactory as it gave erratic results and nonuniform stripping. The first series of tests to study the porosity and uniformity of aluminum coatings on steel have been completed. These tests were based on gas evolution in 5, 15, and 25 per cent nitric acid at 75 and 100 F. Tests in different laboratories at 75 F were in good agreement. Those conducted at 100 F varied greatly. The subcommittee plans to investigate methods for analysis of aluminum coatings on steel.

Subcommittee XI on Sheet Specifications (E. P. Beachum, chairman) prepared the proposed Tentative Specification for Zinc-Coated (Galvanized) Iron or Steel Sheets for Culverts and Underdrains appended to this report. Specifications are being written for galvanized sheets of structural quality and for aluminum-coated sheets.

Subcommittee XII on Wire Specifications (J. F. Occasione, chairman) is presently working on specifications for zinc-coated flat armor tape and for copper-covered steel guy strand.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) recommended the changes in the Standard Specifications for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 59) given earlier in this report. These changes, prepared by a task group, were intended to preclude the use of embrittled iron hardware.

Another task group is presently engaged in an investigation of premature

---

[3] 1958 Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

TABLE I.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at Altoona, Pa., on April 22, 1926.

Sheet[c]	No. 16 Gage, 2.5 oz per sq ft[a]		No. 22 Gage, 2.5 oz per sq ft[a]			No. 22 Gage 2.0 oz per sq ft[a]			No. 22 Gage, 1.5 oz per sq ft[a]			No. 22 Gage, 1.25 oz per sq ft[a]			No. 22 Gage, 0.75 oz per sq ft[a]			No. 28 Gage, 0.75 oz per sq ft[a]			No. 22 Gage, Uncoated
	Years to		Years to			Years to			Years to			Years to			Years to			Years to			
	1st Rust	100 per cent Rust	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perfora tion	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	Years to Perforation
R (T)[d]	...	...	4.3	12.0	21.0	3.3	9.5	18.5	3.3	5.0	14.5	2.3	4.3	14.5	...	...	...	...	...	...	4.3
(B)[d]			4.5	11.5	21.5	3.3	9.0	16.5	3.3	7.0	17.0	2.3	4.5	16.0							
V (T)	4.3	11.0	4.3	9.0	[b]	3.3	7.0	17.5	2.3	7.0	14.5	2.3	5.2	13.5	1.4	3.3	9.0	...	...	...	4.3(V)
(or U) (B)	4.3	7.0	4.8	9.0	17.5	4.3	6.8	19.0	2.5	7.0	15.0	2.3	4.5	15.0	2.3	3.3	13.0				4.3(U)
N (T)	...	...	4.3	8.5	22.5	3.3	8.5	20.0	3.3	5.5	17.0	2.3	4.5	15.0	1.4	2.8	13.0	2.0	3.3	7.0	7.5
(B)			3.5	12.0	24.0	3.5	8.0	22.0	3.3	5.9	19.0	2.3	4.5	[b]	1.4	3.3	15.0	2.3	3.3	11.0	
T (T)	4.5	10.5	5.5	11.5	22.5	3.5	7.5	20.0	2.3	5.0	18.5	2.3	5.0	15.0	1.4	2.8	13.5	2.0	2.8	9.0	8.5
(B)	4.8	9.0	6.3	11.5	[b]	3.5	7.0	22.0	2.5	5.2	17.0	2.3	4.3	17.0	2.3	3.0	15.0	2.3	3.3	11.0	
Y (T)	4.3	11.5	3.5	11.5	*	3.3	8.5	*	2.5	5.0	*	2.3	3.3	*	2.3	3.5	*	1.4	3.5	*	*
(B)	4.5	13.7	3.3	9.5	*	4.3	9.0	*	3.3	5.2	*	2.5	4.3	*	2.3	3.3	*	2.3	3.3	*	
S (T)	4.3	12.0	4.3	12.0	22.0	4.3	8.5	20.5	3.0	4.5	19.0	2.3	4.0	17.0	2.0	2.8	13.5	2.0	3.0	7.0	9.0
(B)	4.3	12.5	4.8	14.5	24.0	4.3	8.5	23.0	3.3	6.3	20.5	3.0	4.3	19.0	2.3	2.8	15.0	2.3	3.0	10.5	
C (T)	...	...	4.0	10.5	26.0	3.3	8.5	22.5	2.5	6.5	21.5	2.3	4.5	19.0	1.4	3.5	[b]	...	...	...	
(B)			5.8	13.5	26.0	4.3	10.0	24.0	3.3	6.5	19.0	2.5	4.3	19.5	1.4	3.0	[b]				
K (T)	4.3	12.5	4.3	11.5	25.0	4.3	7.5	24.0	3.3	5.2	21.5	2.5	4.3	[b]	2.3	3.0	[b]	2.3	2.8	10.5	13.0
(B)	4.3	11.5	4.5	11.5	32.0	4.3	8.5	27.0	3.3	5.0	25.0	3.3	4.3	21.0	2.3	3.0	21.0	2.3	3.0	10.5	
W (T)	...	...	5.2	10.5	21.5	3.5	6.5	19.0	3.3	5.0	15.0	2.3	4.0	14.5	...	...	...	...	...	...	5.0
(B)			5.2	12.0	23.0	4.3	7.5	19.5	3.3	5.5	17.0	2.3	4.0	15.0							
Average	4.4	11.1	4.6	11.2	...	3.8	8.1	...	3.0	5.7	...	2.4	4.3	...	1.9	3.1	...	2.1	3.1	...	

* Data not recorded because of uncertain composition of steels.

[a] Coating weight is "oz per sq ft" of sheet (both sides).

[b] Blown away or discarded due to mechanical damage.

[c] Compositions of individual materials given in 1927 Report of Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 27, p. 175 (1927).

[d] "T" refers to the front end of sheet as it went through the galvanizing pot, "B" refers to the back end, see 1927 Report of Committee A-5, *ibid.*

TABLE II.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.

Exposed at State College, Pa., on April 23, 1926.

Sheet[b]	No. 16 Gage, 2.5 oz per sq ft[a]		No. 22 Gage, 2.5 oz per sq ft[a]			No. 22 Gage, 2.0 oz per sq ft[a]			No. 22 Gage, 1.5 oz per sq ft[a]			No. 22 Gage, 1.25 oz per sq ft[a]			No. 22 Gage, 0.75 oz per sq ft[a]			No. 28 Gage, 0.75 oz per sq ft[a]			No. 22 Gage Uncoated
	Years to		Years to			Years to			Years to			Years to			Years to			Years to			
	1st Rust	100 per cent Rust	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	Years to Perforation
R (T)	24.0	*	26.0	*	*	19.0	*	*	17.5	*	*	14.5	*	*	c			c			26.0
(B)	27.0	*	29.0	*	*	20.0	*	*	16.5	*	*	15.0	*	*	c			c			
V (T)	27.0	*	21.5	*	*	18.5	*	*	16.0	*	*	15.0	*	*	9.5	23.0	*	c			24.0(V)
(or U) (B)	24.0	*	29.0	*	*	25.0	*	*	15.5	*	*	16.5	*	*	10.5	24.0	*	c			25.0(U)
N (T)	c		20.5	*	*	22.5	*	*	17.5	*	*	12.0	*	*	6.5	20.5	*	11.0	23.0	*	28.0
(B)	c		28.0	*	*	22.5	*	*	15.5	*	*	13.5	*	*	9.0	23.0	*	11.5	30.0	d	
T (T)	27.0	*	*	*	*	21.0	*	*	16.0	*	*	15.0	*	*	9.0	26.0	*	9.0	24.0	*	*
(B)	31.0	*	*	*	*	23.0	*	*	14.5	*	*	13.5	*	*	10.5	22.5	*	12.0	25.0	*	
Y (T)	21.5	*	22.5	*	*	19.5	*	*	17.5	*	*	14.5	*	*	10.0	22.5	*	9.5	24.0	*	25.0
(B)	22.5	*	29.0	*	*	19.0	*	*	18.5	*	*	14.5	29.0	*	10.5	30.0	*	13.0	26.0	*	
S (T)	22.0	*	28.0	*	*	25.0	*	*	19.0	*	*	14.5	32.0	*	10.5	21.5	*	11.5	22.0	*	*
(B)	22.5	*	32.0	*	*	28.0	*	*	19.5	*	*	15.5	30.0	*	11.0	21.5	*	11.5	21.5	*	
C (T)	c		22.5	*	*	17.5	*	*	16.0	*	*	14.5	*	*	9.5	24.0	*	c			*
(B)	c		*	*	*	21.5	*	*	18.5	*	*	14.5	30.0	*	10.0	24.0	*	c			
K (T)	23.0	*	25.0	*	*	22.5	*	*	17.5	*	*	14.5	31.0	*	11.5	24.0	*	10.5	23.0	*	*
(B)	24.0	*	30.0	*	*	28.0	*	*	21.0	*	*	16.5	33.0	*	11.5	22.5	*	11.5	21.0	*	
W (T)	c		29.0	*	*	25.0	*	*	16.5	33.0	*	13.5	31.0	*	c			c			27.0
(B)	c		*	*	*	28.0	*	*	15.0	*	*	14.5	30.0	*	c			c			
Average	24.6	...	...	...	...	22.5	...	...	17.1	...	...	14.6			10.0	23.5		11.1	24.0		

* Data not yet available.

[a] Coating weight is "oz per sq ft" of sheet (both sides).

[b] (T) = Corrugated sample from top of sheet on west side of rack with bottom surface upward.
(B) = Corrugated sample from bottom of sheet on east side of rack with top surface upward.

[c] There were no specimens exposed.

[d] Reported as perforation rather than 100 per cent rust in 1956 *Proceedings*.

corrosion of galvanized steel. Six participating metallurgists reported on the chemical composition of the steel and on the composition of the coating on an angle supplied by a power company in Ohio. The analysis of the steel under study is of the semi-killed type conforming to Specification A 7. From the reports of the investigators it is apparent that a change in these specifications to limit the silicon content does not appear to be necessary. However, a study will be made on bars and angles of steel falling within Specification A 7 but made by four regular steel-making methods: rimmed, capped, semi-killed, and fully-killed.

Subcommittee XIV on Sheet Tests (C. P. Larrabee, chairman) inspected the atmospheric exposure tests of corrugated sheets at Altoona on April 17, 1958, and at State College on April 17, 1958 and April 21, 1959. The report of both inspections follows:

At Altoona, Pa., the copper-steel sheet K, which originally had 2.5 oz per sq ft of zinc coating, was found perforated on April 17, 1958 after 32 years of exposure, thus finishing the Altoona phase of the investigation. The complete and final record of the test at Altoona is shown in Table I. A comparison of the data in this table and those in Table II (State College, Pa.) illustrates the tremendous differences in corrosion rates of galvanized steels that can occur even though the exposure sites are only about 35 miles apart.

In the fall of 1960, a task force of the subcommittee expects to expose sheets representing six continuous galvanizing lines and four conventional galvanizing pots in the ASTM test sites at State College, Pa.; Newark, N. J.; Kure Beach, N. C.; Brazos River (near Freeport, Tex.); and Point Reyes, Calif. (north of San Francisco). Sheets of aluminum-coated steels will also be exposed.

Subcommittee XV on Wire Tests (F. M. Reinhart, chairman) inspected the 1936 atmospheric exposure test program of wire and wire products at Bridgeport, Conn., on April 20, 1959, and at State College, Pa., on April 21, 1959. The wires at the other sites were inspected by the university inspectors at each site during the autumn of 1959.

A 20-yr report on the 1936 wire exposure tests is in preparation and nearing completion.

A task force has planned a new exposure test of aluminum-coated wire and wire products.

Subcommittee XVI on Hardware Tests (W. W. Bradley, chairman) prepared, under the direction of former chairman A. Mendizza, the report on Field Tests of Atmospheric Corrosion of Hardware – 1929 Program and 1958 Program, which is appended to this report.[5]

This report has been submitted to letter ballot of the committee, which consists of 109 members; 67 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

HENRY F. HORMANN,
Chairman.

C. P. LARRABEE,
Secretary.

---

[5] See p. 116.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-5 presented to the Society through the Administrative Committee on Standards the recommendation that Specifications for Zinc Coated (Galvanized) Steel Sheets of Structural Quality, Coils and Cut Lengths be published as tentative. This recommendation was accepted by the Standards Committee on September 29, 1960, and the new tentative specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 1, bearing the designation A 446 – 60 T.

APPENDIX

REPORT OF SUBCOMMITTEE XVI ON FIELD TESTS OF ATMOSPHERIC CORROSION OF HARDWARE

1929 Program and 1958 Program

This report covers the 1959 inspections of the 1929 program at State College, Pa., and that of the new program started in 1958 at three other test locations.

The last reports of the discontinued test sites of the 1929 program appear in the following ASTM *Proceedings*:

Altoona and Brunot Island, Pa.—1944 (Vol. 44), and

Sandy Hook, N. J., and Key West, Fla.—1952 (Vol. 52).

The data from the inspection at State College are shown in Table I. There was no inspection in 1958 at this site. The rate of corrosion at this site is very slow. Aluminum-coated specimens are for the most part still unaffected after 29.6 yr. Not much change has been observed on the other coatings from the last reported inspection (1957). Certain anomalies have been noted in this and former years' inspections with regard to the performance of electroplated zinc and hot-dip zinc coatings. For instance, the 1-in. tubular goods with a 1.62-oz coating of electrodeposited zinc show no rusting as against slight rusting and yellow rust staining for the specimen with the 1.83-oz coating of hot-dip zinc. Also, some nipple and ell specimens with comparatively thicker electroplated zinc coatings are reported as having more rust than specimens plated with thinner coatings. It is believed that these apparent anomalies are probably due to errors in the original thickness measurements.

The specimens of the 1958 program were inspected after a one-year exposure at the three sites: Newark, N. J.; Kure Beach (80- and 800-ft sites), N. C. Preliminary data, covering basis alloy compositions, specimen form, specimen identification, coating procedures, and coating thickness measurements are reported in the 1959 ASTM *Proceedings*.

In Tables II to IV are shown condensed data of the ranges and average thickness for all sets of five replicate specimens exposed at the three locations. Discussion of the data in Tables II to IV is given below. In general, there is very little activity to report. In those cases where definite corrosion or rusting has occurred, the specimens in question have been identified in accordance with the numbering system shown on Fig. 2 of the report in the 1959 ASTM *Proceedings*. In this system, a specimen is identified by four numbers, for example 0-5-11-14, where the first number represents the basis alloy, the second the coating, the third the exposure location, and the last is one of five replicate numbers. The test data so far indicate that one of the two hot-dip aluminum coatings on test, coded 7, is beginning to show signs of deterioration in the two marine locations. There is as yet no evidence of influence by basis alloy or specimen form. Where abbreviations have been used they have the same significance as indicated in Table I.

Discussion of Table II

Report of the June 12, 1959 inspection of variously coated carbon and low-alloy

TABLE I.—REPORT OF INSPECTION ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC., EXPOSED TO ATMOSPHERE ON TEST RACKS AT STATE COLLEGE, PA. 1929 PROGRAM.

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; Y, yellow or orange rust; R*, rust spots widely distributed; Tr, trace; TH, threads; C, average of nipple and ell.

Coating	Weight, oz per sq ft of surface	4-21-59 1510 weeks 29.6 yr
3 by 3 by 5/16-in. Plain Steel Angles		
Hot-dip galvanized	2.52	TrY
Hot-dip aluminum	0.89	0
Electric sherardized	1.38	5R-95Y
Lead-plated	...	25R-65R*
3 by 3 by 5/16-in. Copper-Bearing Angles		
Hot-dip galvanized	2.88	TrR-TrY
Hot-dip aluminum	0.82	0
Electric sherardized	1.44	25R-75Y
Lead-plated	...	15R-75R*
2½ by 1½ by 3/16-in. Plain Steel Angles		
Hot-dip galvanized	3.24	TrY
Hot-dip aluminum	0.75	0
Lead-plated	...	15R-70R*
2½ by 1½ by 3/16-in. Copper-Bearing Angles		
Hot-dip galvanized	2.75	TrY
Hot-dip aluminum	0.50	0
Lead-plated	...	30R-65R*
2-in. Diameter Tubular Goods		
Electrogalvanized	1.58	5R
Hot-dip galvanized	2.51	25Y
Hot-dip aluminum	4.73	0
Gas sherardized	0.66	70R-30Y
Lead-plated	...	60R-40R*
1-in. Tubular Goods		
Electrogalvanized	1.62	0
Hot-dip galvanized	1.83	5R-95Y
Hot-dip aluminum	1.83	0
Electric sherardized	0.92	100R
Gas sherardized	0.72	80R-20Y
Calorized	...	5R*-95Y
Lead-plated	...	55R-45R*
Amaloy	...	10R-80R*
Forged Steel Tower Fittings		
Hot-dip galvanized	2.67	80Y
Hot-dip galvanized	1.53	25R-75Y
Hot-dip aluminum	...	0
Electric sherardized	0.50	65R-35Y

TABLE I.—*Continued*

Coating	Weight, oz per sq ft of surface	4-21-59 1510 weeks 29.6 yr
¾-in. Nipples and Ells (C)		
Electrogalvanized MI[a]	1.60	10R
Electrogalvanized CI[b]	2.19	20R-10Y
Electrogalvanized MI	2.65	70R-20Y
Electrogalvanized CI	1.25	50R-30Y
Hot-dip galvanized MI	1.82	15R-80Y
Hot-dip galvanized CI	1.44	15R-80Y
Hot-dip aluminum MI	3.20	RTH
Hot-dip aluminum CI	2.80	RTH
Electric sherardized MI	0.75	40R-60Y
Amaloy MI and CI	...	60R-40R*
Tower Clamps		
Hot-dip galvanized MI	2.00	20R-80Y
Hot-dip galvanized CI	2.38	TrR-50Y
Hot-dip aluminum MI	3.16	0
Hot-dip aluminum CI	2.51	0
1½ by 1½ by ⅛-in. Plain Steel Angles		
Hot-dip galvanized	3.16	TrR-5Y
Hot-dip aluminum	0.89	0
Lead plated	...	10R-70R*
1½ by 1½ by ⅛-in. Copper-Bearing Angles		
Hot-dip galvanized	3.20	TrR-5Y
Hot-dip aluminum	1.11	0
Lead-plated	...	20R-75R*
No. 14 by 2½-in. Copper Bearing Steel Flats		
Hot-dip galvanized	3.20	TrR-TrY
Hot-dip aluminum	0.47	0
Lead plated	...	25R-60R*
Knockout Boxes		
Hot-dip galvanized	1.50	TrR-70Y
Electric sherardized	0.28	5R-95Y
Amaloy	...	50R-50R*
Hot-dip aluminum	1.84	0
Bulldog Clamps		
Hot-dip aluminum	1.35	TrR
¼ by 3-in. Plain Steel Flats		
Hot-dip galvanized	2.49	TrR-5Y
Hot-dip aluminum	0.90	0
Lead-plated	...	10R-85R*

[a] MI = malleable iron.

[b] CI = cast iron.

TABLE I.—*Continued*

Coating	Weight, oz per sq ft of surface	4-21-59 1510 weeks 29.6 year
¼ BY 3-IN. COPPER-BEARING FLATS		
Electrogalvanized	0.84	95R-5Y
Hot-dip galvanized	2.67	TrR-5Y
Hot-dip aluminum	0.66	0
Lead-plated	...	10R-85R*
NO. 792 WINDOW FRAME SECTION		
Hot-dip galvanized	2.60	TrY
Hot-dip aluminum	1.39	0

steels exposed to the atmosphere for 1.0 yr at Newark (New York Area), N. J.

Hot-Dip Zinc

General Observations. — All specimens were satisfactory, both top and underside, excepting specimen 0-5-11-15 which has one large rust spot on top side.

Hot-Dip Aluminum Code 6

General Observations.—

Flats had few pin hole rust spots and a few small lightly stained areas on top surfaces of several panels in all sets. The same, but to a lesser degree on underside.

Bents Many panels had slight imperfection in coating where stain has developed. Otherwise, appearance much like flats.

Rounds were mostly satisfactory. Light stains on two specimens.

Hot-Dip Aluminum Code 7

General Observations.—

Flats were mostly satisfactory. Few pin point rust spots and lightly stained areas on a few panels both top and underside. Some light staining around identification holes.

Bents Most specimens have one or two small lightly stained areas on top side indiscriminately distributed. No evidence of influence by alloy or shape. Undersides show stains around the identification holes and minor staining on vertical sections.

Rounds had light staining at edges on some specimens.

Electroplated Zinc

General Observations. — All specimens were satisfactory, excepting that top surfaces have occasional tiny red stains, believed to be deposited from environment. They do not appear on the underside.

The underside of flat specimen 1-8-11-18 has a rust spot about 2 per cent in area. Round specimen No. 16 (carbon steel) has scattered rust spots, top and underside.

Sprayed Zinc

General Observations. — All specimens were satisfactory.

Sprayed Aluminum

General Observations. — All specimens were satisfactory, excepting light rust staining around identification holes (the holes were not coated), which is more noticeable on underside.

DISCUSSION OF TABLE III

Report of the June 4, 1959 inspection of variously coated carbon and low alloy steels exposed to the atmosphere for 0.94 yr at Kure Beach (800-ft site), N. C.

TABLE II.—NEWARK (NEW YORK AREA), N. J.

Ranges and Averages of Thickness in Mils (0.001 in.) for 5 Replicate Specimens at Each Site.

Basis Alloy	Specimen Form	Hot-Dip Zinc		Hot-Dip Aluminum No. 6		Hot-Dip Aluminum No.7		Electroplated Zinc		Sprayed Zinc and Aluminum Range
		Range	Avg	Range	Avg	Range	Avg	Range	Avg	
C……………………	Flat	1.86 to 2.70	2.28	1.86 to 2.48	2.17	2.19 to 3.70	2.75	2.60 to 3.20	2.76	3.0 to 4.0
	Bent	1.91 to 2.28	2.08	1.95 to 2.58	2.24	2.49 to 2.96	2.69	1.62 to 2.46	1.96	3.0 to 4.0
	Round	4.32 to 5.90	5.08	2.25 to 3.99	3.54	2.98 to 3.78	3.32	1.64 to 2.76	2.53	...
I……………………	Flat	2.81 to 3.74	3.28	1.90 to 2.23	2.04	2.77 to 4.28	3.49	2.88 to 3.53	3.23	3.0 to 4.0
	Bent	2.28 to 2.46	2.39	1.80 to 2.06	1.94	2.16 to 2.49	2.26	2.35 to 3.39	2.92	3.0 to 4.0
	Round	4.21 to 5.53	4.86	2.73 to 3.73	3.36	2.40 to 6.15	4.52	2.29 to 3.35	2.74	...
II……………………	Flat	2.54 to 3.18	2.79	1.87 to 2.57	2.29	2.12 to 4.60	3.06	...	...	...
	Bent	2.41 to 2.72	2.51	1.52 to 1.76	1.64	2.22 to 2.65	2.38	...	...	...
III……………………	Flat	2.30 to 4.19	3.36	2.66 to 4.06	3.22	2.49 to 3.29	2.78	...	...	...
	Bent	2.09 to 2.84	2.33	1.91 to 2.52	2.09	2.21 to 2.42	2.37	...	...	...
	Round	4.05 to 6.00	5.30	3.28 to 4.62	3.85	3.00 to 4.50	3.90	...	...	...
IV……………………	Flat	2.92 to 3.42	3.17	1.77 to 2.51	2.27	2.88 to 3.52	3.45	2.54 to 2.84	2.69	3.0 to 4.0
	Bent	2.01 to 2.35	2.17	1.61 to 1.75	1.68	2.09 to 2.70	2.42	1.81 to 2.93	2.40	3.0 to 4.0
	Round	4.05 to 6.28	4.88	2.34 to 4.61	3.29	2.15 to 4.53	3.62	2.23 to 2.99	2.68	...
NIC[a]……………………	Flat	2.47 to 4.88	4.26	...	...	...	...	...	...	...
NIM[b]……………………	Flat	5.97 to 6.57	6.34	...	...	...	...	...	...	...
MI[c]……………………	Flat	6.89 to 7.43	7.19	...	...	...	...	...	...	...

[a] Nodular iron, cast.

[b] Nodular iron, machined.

[c] Malleable iron.

TABLE III.—KURE BEACH (800-FT SITE), N. C.

Range and Average of Thickness in Mils (0.001 in.) for 5 Replicate Specimens at Each Site.

Basis Alloy	Specimen Form	Hot-Dip Zinc		Hot-Dip Aluminum No. 6		Hot-Dip Aluminum No. 7		Electroplated Zinc		Sprayed Zinc and Aluminum
		Range	Avg	Range	Avg	Range	Avg	Range	Avg	Range
C	Flat	2.01 to 2.37	2.17	2.28 to 3.17	2.61	3.29 to 4.07	3.77	2.14 to 3.13	2.70	3.0 to 4.0
	Bent	1.98 to 2.82	2.20	2.06 to 2.55	2.34	2.34 to 2.86	2.55	1.86 to 3.04	2.38	3.0 to 4.0
	Round	3.12 to 5.02	3.84	2.18 to 5.50	3.83	3.21 to 3.79	3.49	2.11 to 2.59	2.33	...
I	Flat	3.01 to 3.66	3.26	2.06 to 2.74	2.32	3.17 to 3.88	3.57	2.61 to 3.22	2.98	3.0 to 4.0
	Bent	2.49 to 2.74	2.58	1.69 to 1.99	1.85	2.28 to 2.96	2.63	2.61 to 3.69	3.18	3.0 to 4.0
	Round	3.66 to 6.35	5.12	3.22 to 3.86	3.43	2.73 to 5.33	3.68	1.90 to 3.22	2.63	...
II	Flat	2.47 to 3.77	2.89	2.17 to 2.73	2.44	2.87 to 4.71	3.80	...	...	...
	Bent	2.19 to 2.45	2.32	1.42 to 1.96	1.58	1.90 to 3.41	2.46	...	...	...
III	Flat	2.51 to 3.43	2.78	2.38 to 2.79	2.61	2.78 to 4.24	3.61	...	...	...
	Bent	2.76 to 3.32	2.99	1.89 to 2.61	2.23	2.39 to 2.76	2.60	...	...	...
	Round	3.19 to 5.01	4.16	2.44 to 4.65	3.65	3.12 to 4.85	3.71	...	...	...
IV	Flat	2.78 to 3.47	3.05	2.01 to 2.44	2.24	2.40 to 5.03	3.71	1.91 to 2.87	2.50	3.0 to 4.0
	Bent	2.01 to 2.21	2.08	1.72 to 1.98	1.84	2.02 to 2.76	2.33	1.92 to 3.14	2.49	3.0 to 4.0
	Round	4.17 to 5.44	5.01	2.69 to 5.00+	3.48+	1.06 to 4.68	3.65	2.31 to 2.59	2.47	...
NIC[a]	Flat	2.83 to 4.87	4.18	...	...	...	...	...	...	...
NIM[b]	Flat	6.03 to 6.22	6.08	...	...	...	...	...	...	...
MI[c]	Flat	6.94 to 7.66	7.22	...	...	...	...	...	...	...

[a] Nodular iron, cast.

[b] Nodular iron, machined.

[c] Malleable iron.

Hot-Dip Zinc

General Observations. — All specimens were satisfactory, both top and underside.

Bents, specimen 2-7-12-17: 5Y top, 30Y underside. Many others have stained areas of less than 5 per cent of the total surface.

TABLE IV.—KURE BEACH (80-FT SITE), N. C.

Ranges and Averages of Thickness in Mils (0.001 in.) for 5 Replicate Specimens at Each Site.

Basis Alloy	Specimen Form	Hot-Dip Zinc		Hot-Dip Aluminum No. 6		Hot-Dip Aluminum No. 7		Electroplated Zinc	
		Range	Avg	Range	Avg	Range	Avg	Range	Avg
C.........	Flat	2.21 to 2.40	2.30	1.99 to 2.47	2.25	2.28 to 4.70	3.16	1.68 to 2.53	2.19
	Bent	2.09 to 2.21	2.15	1.81 to 2.51	2.32	2.12 to 2.66	2.36	1.72 to 2.66	2.28
	Round	3.86 to 5.56	4.70	2.71 to 3.58	3.10	3.70 to 4.89	4.08	2.38 to 3.15	2.75
I..........	Flat	3.03 to 3.59	3.22	1.96 to 2.70	2.44	3.01 to 3.70	3.59	2.89 to 4.14	3.26
	Bent	2.28 to 2.85	2.62	1.74 to 1.91	1.80	2.21 to 3.31	2.67	1.85 to 3.02	2.70
	Round	3.81 to 5.83	4.69	2.96 to 3.44	3.14	2.67 to 4.78	3.90	2.14 to 3.81	2.71
II.........	Flat	2.62 to 3.08	2.80	2.14 to 3.31	2.54	2.84 to 4.81	3.80	...	...
	Bent	2.14 to 2.56	2.38	1.54 to 1.69	1.60	2.16 to 2.36	2.31	...	...
III........	Flat	3.07 to 3.90	3.52	2.43 to 3.02	2.72	2.69 to 3.61	3.27	...	...
	Bent	2.52 to 3.44	2.89	1.96 to 2.62	2.31	2.54 to 2.81	2.69	...	...
	Round	3.04 to 4.71	4.11	3.34 to 4.17	3.78	3.77 to 4.40	3.99	...	...
IV........	Flat	3.08 to 3.43	3.19	1.91 to 2.77	2.31	3.17 to 3.58	3.40	1.91 to 3.56	2.58
	Bent	2.52 to 3.44	2.89	1.59 to 1.90	1.76	1.69 to 2.04	1.87	2.55 to 3.19	2.92
	Round	4.73 to 5.68	5.21	3.31 to 4.58	3.58	4.07 to 5.61	4.76	2.37 to 3.06	2.76
NIC[a]......	Flat	2.77 to 4.83	4.15	...	...	...	...	...	...
NIM[b].....	Flat	5.51 to 6.57	6.07	...	...	...	...	...	...
MI[c].......	Flat	6.77 to 7.44	7.11	...	...	...	...	...	...

[a] Nodular iron, cast.

[b] Nodular iron, machined.

[c] Malleable iron.

Hot-Dip Aluminum Code 6

General Observations.—

Flats and Bents A few specimens have stains approaching 5 per cent of the surface area.

Rounds All specimens satisfactory.

Hot-Dip Aluminum Code 7

Observations.—

Flats, specimen 2-7-12-14: 5Y top, 15Y underside; specimen 2-7-12-16: <5Y top, 20Y underside. Several other specimens have stains approaching 5 per cent of the surface area.

Rounds, specimen 0-7-12-14: 15Y top, satisfactory underside. Several others have slight rust staining.

Electroplated Zinc

General Observations. — All specimens were satisfactory, both top and underside.

Sprayed Zinc

General Observations. — All specimens were satisfactory, both top and underside.

Sprayed Aluminum

General Observations. — All specimens were satisfactory, excepting some light staining at the identification holes, which were not coated.

DISCUSSION OF TABLE IV

Report of the June 4, 1959, inspection of variously coated carbon and low alloy steels exposed to the atmosphere for 0.94 yr at Kure Beach (80-ft site), N. C.

Hot-Dip Zinc

General Observations. — All specimens were satisfactory, both top and underside.

Hot-Dip Aluminum Code 6

General Observations.—

Flats Several panels have one or two small stained areas both on top and underside. Light staining also around a few holes.

Bents were similar to flats; only few panels affected.

Rounds were all satisfactory.

Hot-Dip Aluminum Code 7

Observations.—

Flats:

Specimen	Topside	Underside
0-7-13-14	5Y	OK
1-7-13-14	5Y	OK
2-7-13-14	20Y	35Y

Several other panels have small stained areas top and underside.

Bents:

Specimen	Topside	Underside
0-7-13-14	20Y	20Y
0-7-13-15	10Y	TrY
0-7-13-16	TrY	OK
0-7-13-17	20Y	10Y
0-7-13-18	TrY	
2-7-13-14	30Y	30Y
2-7-13-16	TrY	5Y
2-7-13-18	20Y	OK
3-7-13-14	5Y	OK

Several other panels have stained areas up to 5 per cent of total surface both on top and underside. Stains appear also at identification holes.

Rounds Two specimens have one small rust spot each. Several others have very little pin hole rusting.

Electroplated Zinc

General Observations. — All specimens were satisfactory, both top and underside.

Respectfully submitted on behalf of the subcommittee,

A. MENDIZZA,
Chairman.

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES*

Committee A-6 on Magnetic Properties held two general meetings during the year: on June 23, 1959, during the Annual Meeting of the Society in Atlantic City, N. J., and on Feb. 2, 1960, in Chicago, Ill., during ASTM Committee Week.

The committee records with sorrow the sudden deaths on Oct. 2, 1959 of J. H. Schneider of the Elliott Co., Ridgeway, Pa., and on Jan. 1, 1960 of C. A. Maynard of the Indiana Steel Products Co., Valparaiso, Ind. Mr. Maynard had long been active in the Task Group working on the Manual of Magnetic Testing which includes nonstandard methods and other magnetic data.

The membership of the committee has risen during the year since there are 14 new members, of whom 4 are replacements for previous members.

The officers elected for the ensuing term of two years are as follows:

Chairman, A. C. Beiler.
Vice-Chairman, D. C. Dieterly.
Secretary, W. S. Eberly.

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Methods of Test for Permeability of Feebly Magnetic Materials (A 342 – 53):[1] Revise as appended hereto.[2]

Standard Method of Test for Alternating Current Core Loss and Permeability of Magnetic Materials (A 343 – 54):[1] Revise as appended hereto.[2]

Standard Methods of Test for Electrical and Magnetic Properties of Magnetic Materials (A 344 – 52):[1] Revise as appended hereto.[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (I. L. Cooter, chairman) has been revising Definitions A 340 and has attempted to maintain uniformity between ASTM definitions and the American Standard definitions sponsored by ASA Sectional Committee C42.

Subcommittee II on Methods of Sampling and Test (J. W. Hale, chairman) has completed work on the interlaminar resistance and lamination factor test methods through the activity of a task group under A. E. Kettner, and has incorporated them in the revision of Methods A 344 appended to this report.[2] The subcommittee has also revised Methods

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 3.

[2] The revised methods appear in the 1960 Supplement to Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A 342 and A 343, as appended to this report.[2] Task Groups on Density of Aluminum-Iron Alloys and on Effect of Aluminum Additions on Silicon Steel Density have accumulated data for a forthcoming revision of portions of Method A 34.

The following additional projects under active task groups indicate the scope of work currently in progress: manual of testing, magnetic amplifier core materials, tests for cut tape wound cores, for magnetic shielding, d-c tests of high coercive magnets, high-frequency Epstein testing, and testing iron-nickel and high permeability alloys.

This report has been submitted to letter ballot of the committee, which consists of 35 voting members; 28 members returned their ballots, of whom 26 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. C. BEILER,
Chairman.

W. S. EBERLY,
Secretary.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS*

During the year, Committee A-10 held two meetings: on June 23, 1959, during the Annual Meeting of the Society in Atlantic City, N. J., and on Feb. 1 and 2, 1960, during ASTM Committee Week in Chicago, Ill.

The committee has a total membership of 115, of whom 52 are classified as producers, 42 as consumers, and 21 as general interest members.

A new Subcommittee II has been formed for the purpose of supervising the editorial quality of the committee's standards. This group is under the chairmanship of A. G. Cook.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. L. Wyman.
Vice-Chairman, M. A. Cordovi.
Secretary, G. R. Woodrow.

REVISION OF TENTATIVE

The committee recommends that the Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (**A 312 - 59 T**)[1] be revised as indicated in the 1960 Report of Committee A-1 on Steel.[2]

REVISIONS OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Tentative Specifications for Hot-Rolled and Cold-Finished Corrosion-Resisting Steel Bars (A 276 - 59):[2]

Table II.—For all 300 types, for the annealed condition, cold-finished, and over ½ in. in thickness, change the value for elongation in 2 in. from "40" to "35."

Standard Specifications for Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 55):[2]

Table II.—For all grades reduce the sulfur and phosphorus contents from the present "0.05" per cent to "0.04" per cent, max. Add grades CF-3, CF-3M, and CG-8M, with the chemical requirements shown in the accompanying Table I. The carbon content of grade CF-3 is to be subject to the rounding-off method of the Society in Recommended Practice E 29.

Table III.—Add grades CF-3, CF-3M, and CG-8M, with the tensile property requirements shown in the accompanying Table II. Change the tensile strength of grade CF-8 from the present "70,000" to read "65,000" psi, min.

Standard Specifications for Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 - 55):[3]

Table I.—For all grades reduce the

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1959 Supplement to Book of ASTM Standards, Part 1.

[2] This revision has been recommended by a Joint A-1 and A-10 Special Subcommittee on Stainless Steel Tubing. It appears in the 1960 Supplement to Book of ASTM Standards, Part 1.

[3] 1958 Book of ASTM Standards, Part 1.

phosphorus and sulfur contents from the present "0.05" per cent to "0.04" per cent, max. Add grades HD, HL, and HN, with the chemical requirements shown in the accompanying Table III.

Table II.—Add grades HD, HL, and HN, with the tensile property requirements shown in the accompanying Table IV.

TABLE I.—ADDITIONS TO TABLE II, SPECIFICATIONS A 296.

Grade	Carbon, max, per cent	Manganese, max, per cent	Silicon, max, per cent	Phosphorus, max, per cent	Sulfur, max, per cent	Chromium, max, per cent	Nickel, max, per cent	Molybdenum, max, per cent
CF-3........	0.03	1.50	2.00	0.04	0.04	17 to 21	8 to 12	...
CF-3M......	0.03	1.50	1.50	0.04	0.04	17 to 21	9 to 13	2.0 to 3.0
CG-8M.....	0.08	1.50	1.50	0.04	0.04	18 to 21	9 to 13	3.0 to 4.0

TABLE II.—ADDITIONS TO TABLE III, SPECIFICATIONS A 296.

Grade........................	CF-3	CF-3M	CG-8M
Tensile strength, min, psi.	65 000	70 000	75 000
Yield strength, min, psi..	28 000	30 000	35 000
Elongation in 2 in., min, per cent.............	35	30	25

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as set forth in the Annual Report of Committee A-1 on Steel, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for:

Seamless and Welded Ferritic Stainless Steel Tubing for General Service (**A 268 – 58**),[3]

Seamless and Welded Austenitic Stainless Steel Tubing for General Service (**A 269 – 59**),[3]

Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (**A 270 – 58**),[3] and

Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (**A 271 – 59**).[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Recommended Practice for:

Boiling Nitric Acid Test for Corrosion-Resisting Steels (**A 262 – 55 T**),[4] and

Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel (**A 393 – 55 T**).[4]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II, Editorial (A. G. Cook, chairman) has been established during the year and has already taken several steps in the direction of obtaining editorial uniformity in the recommendations proposed by Committee A-10 and is laying the groundwork for a comparable review of existing standards.

Subcommittee IV on Methods of Corrosion Testing (O. B. Ellis, chairman.)—The report of the joint ASTM-NACE task group on stress-corrosion cracking of austenitic stainless steels is being published as *ASTM STP 264*. This report includes a number of case histories together with an up-to-date annotated bibliography on stress-corrosion cracking.

---

[4] 1958 Book of ASTM Standards, Part 3.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Work is continuing on the investigation of the nitric-hydrofluoric acid test and the boiling sulfuric acid-ferric sulfate test for intergranular corrosion of type 316 L stainless steel. Preliminary data indicate that the nitric-hydrofluoric acid test may have considerable merit for the evaluation of type 316 L stainless steel.

The subcommittee recommended the continuation of Recommended Practices A 262 and A 393 as tentative because work is continuing on both of these methods. It recommended that Method A 279 be adopted as standard.

TABLE III.—ADDITIONS TO TABLE I, SPECIFICATIONS A 297.

Grade	Carbon, max, per cent	Manganese, max, per cent	Silicon, max, per cent	Sulfur, max, per cent	Phosphorus, max, per cent	Chromium, max, per cent	Nickel, max, per cent	Molybdenum, max, per cent
HD	0.50	1.50	2.00	0.04	0.04	26 to 30	4 to 7	0.50
HL	0.20 to 0.60	2.00	2.00	0.04	0.04	28 to 32	18 to 22	0.50
HN	0.20 to 0.50	2.00	2.00	0.04	0.04	19 to 23	23 to 27	0.50

Subcommitte V on Mechanical Tests (R. H. Heyer, chairman).—During the past year Subcommittee V activity has been principally in two fields. The Task Group on Hardness Testing has under way a round-robin test to establish Brinell-Rockwell conversions for austenitic grades. The data from the test results could lead to recommendations for changing certain hardness limits in product specifications where the hardness is given in both Brinell and Rockwell scales.

The Task Group on Tension Testing has accumulated information from a number of laboratories on current practices for tension testing of stainless steels in foil thicknesses. A round-robin program for testing 1 mil thick foil has been set up but has not yet been activated.

Subcommittee VI on Metallography (Russell Franks, chairman).—The work of Subcommittee VI has been devoted primarily to two problems, namely, metallographic identification of finely divided carbides in stainless steel structures containing substantial quantities of sigma phase, and identification of the constituents causing cracking in the columbium-bearing 18-8 steels used in steam power plant installations. Task groups assigned to each problem are examining samples of both types of steels in an attempt to identify these constituents.

TABLE IV.—ADDITIONS TO TABLE II, SPECIFICATIONS A 297.

Grade	HD	HL	HN
Tensile strength, min, psi	75 000	65 000	63 000
Yield strength, min, psi	35 000	35 000	...
Elongation in 2 in., min, psi	8	10	8

Another problem under consideration by the subcommittee relates to the identification of delta ferrite and retained austenite in certain high-temperature metals now being used in jet aircraft engines.

Subcommittee VIII on Specifications for Wrought Products (R. B. Gunia, chairman).—A letter ballot of Committee A-10 indicated overwhelmingly that a general standard for stainless steel wire should be prepared. A task group will be appointed to write the standard.

The subcommittee voted to adopt

Specifications A 429 as standard. It is also preparing a proposed change in the properties of certain bar sizes in Specifications A 276.

Subcommittee X on Specifications for Castings (H. C. Templeton, chairman) has been active in proposing the addition of several new grades of alloys to existing specifications, and in recommending several changes in chemical composition and mechanical requirements in existing grades.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman).—Considerable effort has been expended to define clearly the requirements of heat treating austenitic stainless steel tubing. A proposed rewording of this section of the various specifications has been submitted and is awaiting the outcome of a letter ballot.

The "H" grades of austenitic steels, expressly intended for high-temperature service, have been added to various specifications for stainless steel products. Consideration is being given to the preparation of specifications to separate the products used for "chemical" service and for "high-temperature" service. The subcommittee has also considered the duplication of compositions in Specification A 430.

Discussion of decimal places in reporting chromium and nickel contents in the stainless tubing specifications continues without a resolution. A proposal to increase the silicon content of the austenitic grades from 0.75 to 1.00 per cent maximum resulted in considerable discussion relating to confusion from previous actions. A task force was established to study this matter of silicon content and to come up with a definite proposal for action at the June meeting.

Subcommittee XII on Specifications for High-Temperature, Super-Strength Alloys (W. F. Simmons, chairman) has continued work on specifications for three classifications of alloys—hot-cold-worked alloys, precipitation-hardened alloys, and cast alloys. A final draft of the specification for precipitation-hardened alloys has been prepared and is now ready for letter ballot.

The subcommittee is in the process of revising *ASTM STP No. 170-A*, Compilation of Chemical Compositions and Rupture Strengths of Super-Strength Alloys.

Subcommittee XIII on Specifications for Nuclear Reactor Structural Materials (M. A. Cordovi, chairman).—Five of the six task groups of the subcommittee reported that their review of proprietary specifications dealing with materials for nuclear applications had been completed. The recommendations of these task groups will be formally presented to the cognizant subcommittees of Committee A-10 for their consideration and action. It does not appear likely that embodiment of the task group recommendations will require generation of new specifications for any of the stainless steel products currently under Committee A-10 jurisdiction.

This report has been submitted to letter ballot of the committee, which consists of 115 members; 102 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. Wyman,
Chairman.

G. R. Woodrow,
Secretary.

REPORT OF COMMITTEE B-1
ON
WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: in Washington, D. C., on Oct. 27, 1959; in New York, N. Y., on Jan. 19, 1960; and in Boston, Mass., on April 12, 1960. Meetings of Subcommittees II, IV, and VII also were held during the year.

The committee took note of the impending retirement on July 1, 1960, of C. A. Jordan, a long-time member of the committee who has contributed a great deal to the effectiveness of the committee's work. Accordingly action was taken to elect him an Honorary Member of the committee.

The committee consists of 87 members, of whom 44 are classified as producers, 28 as consumers, and 15 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, D. Halloran.

Vice-Chairman, C. E. Topping.

Secretary, A. A. Jones.

Members-at-Large, Advisory Subcommittee: D. R. Brobst, L. B. Curtis, A. A. Defoe, M. F. W. Heberlein, and E. Mark Wolf.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and definitions as appended hereto:[1]

Tentative Specification for Nickel-Coated Soft or Annealed Copper Wire:

With the continuing trend to higher operating temperatures, particularly in electronics and military applications, there has been growing need for industry standardization of conductors for these applications. Accordingly, the new proposed specification for nickel-coated copper wire is recommended to supplement the Specifications for Silver-Coated Soft or Annealed Copper Wire (**B 298 – 56 T**).[2]

Tentative Definitions of Terms Relating to Uninsulated Metallic Electrical Conductors:

During the past decade there has been considerable expansion in the types of metallic electrical conductors available to and used by industry. This has brought new terms and conductor designations into widespread use. The committee has felt a need for suitable definitions covering the various terms used in the specifications under its jurisdiction. Accordingly it recommends the publication of this Glossary as tentative, including the 22 definitions on which the committee so far has reached agreement.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 2.

[2] 1958 Book of ASTM Standards, Part 2.

Tentative Specifications for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (ACSR) (B 232 – 59 T):[3]

Section 4(a).—Add at the end of this section: "(Note 9)."

Explanatory Notes.—Add a new Note 9 to read as follows:

NOTE 9.—The behavior of properly spaced wire joints in stranded conductors is related to both their tensile strength and elongation. Because of its higher elongation properties, the lower strength electric butt weld gives equivalent over-all performance to that of a cold pressure weld in stranded conductors.

TABLE I.—ADDITIONS TO TABLE II, SPECIFICATIONS B 286.

Size Designation	Conductor Construction		Calculated Cross-Sectional Area, cir mils	Theoretical Average Diameter, in.	Length of Lay, in. (Note 2)	D-C Resistance at 20 C, ohms per 1000 ft, max (Note 1)	
	Number of Wires	Nominal Diameter of Each Wire, in.				Annealed Tin or Lead-Alloy Coated	Annealed Silver Coated
49/0.0142..........	49	0.0142	9 880	0.128		1.21	1.14
133/0.0113..........	133	0.0113	16 983	0.170		0.702	0.661
133/0.0142..........	133	0.0142	26 818	0.213		0.445	0.419
133/0.0179..........	133	0.0179	42 615	0.269		0.280	0.263

Tentative Specifications for Copper Conductors for Use in Hookup Wire for Electronic Equipment (B 286 – 59 T):[3]

Section 5(c).—Revise the beginning of the third sentence to read: "For rope-lay-stranded conductors size designation 8-168 and larger, and size 49/0.0142, the length of lay. . . ."

Table II.—Add to this table in proper order as to cm area four sizes of conductor as shown in the accompanying Table I.

Table III.—Add to this table in proper order as to cm area four sizes of conductor as shown in the accompanying Table II.

Explanatory Note 2.—In the second paragraph under f = stranding lay factor, for $f = 1.04$ change the second sentence to read: "Conductors in sizes 8 Awg through 2 Awg incl, and size 49/0.0142 are assumed to be made up of 7 bunched members."

Tentative Specifications for Aluminum-Coated (Aluminized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 341 – 59 T):[3]

Table V.—Delete size 0.1085 in. MW 200-lb coil.

Tentative Method of Test for Electrical Conductivity by Use of Eddy Currents (B 342 – 59 T):[4]

Section 2(a).—Revise to read as follows:

(*a*) Conductivity shall be determined with an instrument that indicates the resistance of a material to the flow of eddy current (see Appendix). The instrument shall be of such design that eddy currents are induced in the test specimen by means of a coil located in a detector tip. The apparent impedance of the coil, which varies with the electrical conductivity of the specimen being tested, is measured (by a balanced bridge circuit or otherwise) in such a manner that the indicating dial or meter may be calibrated to indicate the per cent conductivity (IACS) of the metal.

Section 3.—Add the following sentence at the end of this section: "The thickness shall not be less than that recommended by the instrument manufacturer."

---

[3] 1959 Supplement to Book of ASTM Standards, Part 2.

[4] 1959 Supplement to Book of ASTM Standards, Part 3.

Section 4(a).—Revise to read as follows:

(*a*) The instrument, prior to making the tests, shall be allowed to warm up for a minimum period of time, in accordance with the recommendations of the instrument manufacturer.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following method of test and specifications be approved for reference to letter ballot of the Society for adoption as standard without revision:

TABLE II.—ADDITIONS TO TABLE III, SPECIFICATIONS B 286.

Size Designation	D-C Resistance at 20 C, ohms per 1000 ft, max	
	Annealed Tin or Lead-Alloy Coated	Annealed Silver Coated
TYPE II (STRANDED CONDUCTORS)		
49/0.0142	1.26	1.19
133/0.0113	0.730	0.687
133/0.0142	0.463	0.436
133/0.0179	0.291	0.274

Tentative Method of Test for:

Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication (**B 279 – 53 T**).[5]

Tentative Specifications for:

Silver-Coated Soft or Annealed Copper Wire (**B 298 – 56 T**),[2]

Aluminum Wire for Communication Cable (**B 314 – 57 T**),[2] and

Half-Hard Aluminum Wire for Electrical Purposes (**B 323 – 58 T**).[2]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specification for Rectangular and Square Bare Aluminum Wire for Electrical Conductors (**B 324 – 58 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard with revision as indicated:

Section 9(d).—Revise to read as follows:

(*d*) The wire shall have rounded corners or rounded edges as specified in Table IV and as shown in Fig. 1. Where rounded corners are required the corners of the wire shall be rounded within the limits of radii 25 per cent under and 25 per cent over (as determined by a radius gage) those values specified in Table IV.

TABLE III.—ADDITIONS TO TABLE II, METHOD B 193.

Material	Density, δ, at 20 C, g per cu cm	Temperature Coefficient of Resistance, α, at 20 C
Silver-Coated Copper, ASTM B 298:		
Class A	8.91	0.004
Class B	8.93	0.004
Class C	8.95	0.004
Class D	8.99	0.004
Nickel-Coated Copper, ASTM B 000:		
Class 4	8.89	0.004
Class 10	8.89	0.004
Class 27	8.89	0.004

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 59):[2]

Section 11.—Revise to read as follows:

11. The area of cross-section of the completed conductor shall be not less than 98 per cent of the area indicated in column 1 of Table II.

---

[5] 1958 Book of ASTM Standards, Part 3.

Unless otherwise specified by the purchaser, the manufacturer may have the option of determining the cross-sectional area by either of the following methods, except that in case of question regarding area compliance, Method (*2*) shall be used.

(*1*) The area of cross-section of a conductor may be determined by calculations from diameter measurements, expressed to four decimal places, of its component wires at any point when measured perpendicularly to their axes.

(*2*) The area of cross-section of a conductor may be determined by the Method for Determination of Cross-Sectional Area of Stranded Conductors (ASTM Designation: B 263). In applying that method, the increment in weight resulting from stranding may be the applicable value specified in Section 10(*a*) or may be calculated from the measured component dimensions of the sample under test. In case of question regarding area compliance, the actual weight increment due to stranding shall be calculated.

Standard Method of Test for Resistivity of Electrical Conductor Materials (B 193 – 59):[2]

Table II.—Insert two new groups of data between "Copper" and "Bronze" as shown in the accompanying Table III.

Standard Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (B 230 – 58):[2]

Section 5(c).—Revise to read as follows:

(*c*) When requested by the purchaser, tension tests of specimens containing joints made in the finished wire or during the final drawing, as permitted in Section 11(*b*), shall be made. Such tests shall show the tensile strength to be not less than 11,000 psi for electrical butt-welded joints, and not less than 21,000 psi for cold pressure-welded joints.

Standard Specifications for Concentric-Lay-Stranded Aluminum Conductors, Hard, Three-Quarter-Hard, and Half-Hard (B 231 – 59):[2]

Section 4.—Add at the end of this section: "(Note 7)."

Explanatory Notes.—Add a new Note 7 to read as shown above for new Note 9 in Tentative Specifications B 232.

Standard Specifications for Rolled Aluminum Rods (EC Grade) for Electrical Purposes (B 233 – 55):[2]

Section 1(b).—Add a note at the end of this section to read as follows: "NOTE.—Type EC-H12-B rod is rod of a quality capable of being drawn into wire of 0.0259 in. diameter and smaller."

Standard Specifications for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 – 59):[3]

Table VI.—Delete the size 0.1085 in. MW 200-lb coil.

Standard Specifications for Zinc-Coated (Galvanized) Steel Core Wire (with Coatings Heavier than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 – 59):[3]

Table VI.—Delete the size 0.1085 in. MW 200-lb coil.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of specifications.

Subcommittee II on Methods of Test and Sampling Procedures (R. H. Lloyd, chairman) completed revision of Specification B 342 as noted earlier in the report to avoid restriction to a single manufacturer's instrument. A task group is reconsidering suitable methods of test for determining softness of copper wire.

---

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Conductors of Copper and Copper Alloys (M. V. Yokelson, chairman) completed preparation of the proposed Tentative Specification for Nickel-Coated Soft or Annealed Copper Wire appended to this report. It also completed revision of Specification B 8 as noted above to clarify the method of area determination and bring it into agreement with similar requirements in B 231.

Task groups are cooperating with the U. S. Army Signal Corps Engineering Laboratories in the study of gray-tin transformation; investigating means for measuring wire diameters with a precision greater than 0.1 mil (that is, in hundredths of a mil) and considering the degree of significance needed in specifying fine wire sizes as a preliminary to establishing diameter tolerances for very fine wires; and reviewing possible need for revision or expansion of tensile properties requirements for alloy wires (Specifications B 105).

Subcommittee VI on Composite Conductors of Copper and Steel (C. E. Ambelang, chairman) has task groups working on the preparation of proposed tentative specifications for Type M copper-covered steel strand including resistivity requirements, and aluminum-clad steel (Alumoweld) both solid wire and stranded.

Subcommittee VII on Conductors of Light Metals (A. L. Kolb, chairman) completed revision of Specifications B 230 as noted above to include strength requirements for electrical butt-welded and cold pressure-welded joints in finished aluminum wire. It also completed clarifying revisions, also noted above, of Specifications B 231, B 232, B 233, B 245, B 261, B 324, and B 341.

Task groups are reconsidering the inspection requirements in the various aluminum rod and wire specifications; considering establishing tensile strength requirements for cold pressure-welded joints in half-hard and three-quarter-hard aluminum wire; and investigating the effects of various in-process welds on finished steel core wire properties.

Joint Task Groups are considering the preparation of standard test report forms for bare electrical conductors; and are considering additional definitions for future inclusion in the new Glossary of Terms, the initial issue of which is appended hereto.

This report has been submitted to letter ballot of the committee, which consists of 87 members; 65½ members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. HALLORAN,
Chairman.

A. A. JONES,
Secretary.

5

REPORT OF COMMITTEE B-2
ON
NON-FERROUS METALS AND ALLOYS*

Committee B-2 on Non-Ferrous Metals and Alloys met on June 24, 1959, in Atlantic City, N. J. Meetings of the Advisory Committee were held on June 23, 1959, in Atlantic City, and on Jan. 14, 1960, in Norristown, Pa.

The committee consists of 146 members, of whom 64 are classified as producers, 54 as consumers, and 28 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman: Alfred Bornemann.

Vice-Chairmen: A. M. Bounds and M. F. W. Heberlein.

Secretary: C. K. Conard.

The following appointments were made during the year: Warren J. Jenkins as secretary of Subcommittee I; C. F. Crumbley as secretary of Subcommittee VII, replacing E. R. Patton; A. F. Jones as chairman of Subcommittee IX, replacing T. W. Lippert; and W. E. Lusby, Jr. as secretary of Subcommittee IX. In connection with the work of Committee E-1 on Methods of Testing, A. M. Bounds was appointed to Subcommittee 24; A. F. Jones to Subcommittee 30, F. L. Moffet to Subcommittee 3, and C. S. DuMont to Subcommittee 4. J. E. Cunningham was appointed to Subcommittee II of Committee E-10 on Radioisotopes and Radiation Effects, and C. D. Coxe was appointed to Subcommittee I of Committee E-11 on Quality Control of Materials.

Subcommittee VIII sponsored a Symposium on Newer Metals at the Third Pacific Area National Meeting of the Society in San Francisco, Calif., October, 1959, with R. I. Jaffee as chairman of the symposium. The papers presented at this symposium will be issued as *ASTM STP No. 272.*

The Secretariat of the ISO Committee 26 on Copper has rested in the United States for some years. However, unless more financial aid is obtained, it will be necessary to relinquish the secretariat.

The committee is recommending that participation in the work of the ISO Committee 18 on Zinc be on the basis of an observer.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Titanium and Titanium Alloy Bars and Billet (B 348 - 59 T),

Zirconium Sponge and Other Forms of Virgin Metals for Nuclear Applications (B 349 - 60 T),

Zirconium and Zirconium Alloy Ingots for Nuclear Application (B 350 - 60 T),

Hot-Rolled and Cold-Finished Zirconium and Zirconium Alloy Bars, Rod, and Wire for Nuclear Application (B 351 - 60 T),

Zirconium and Zirconium Alloy Sheet, Strip, and Plate for Nuclear Applications (B 352 - 60 T), and

---

*** Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.**

Wrought Zirconium and Zirconium Alloy Seamless and Welded Tubes for Nuclear Service (B 353 - 60 T).

Revision and Reversion to Tentative of Standard Specifications for:

White Metal Bearing Alloy (Known Commercially as "Babbitt Metal") (B 23 - 49).

The new Tentative Specification B 348 was accepted by the Standards Committee on September 15, 1959, and it appears in the 1959 Supplement to Book of ASTM Standards, Part 2. The revision of Standard Specification B 23 was accepted on November 6, 1959, and the new tentative specifications relating to zirconium for nuclear applications were accepted on April 14, 1960; they are available as separate reprints.

AMERICAN STANDARDS

The following specifications under the jurisdiction of Committee B-2 were approved by the American Standards Assn. as American Standard on December 15, 1959:

Standard Specifications for:

Slab Zinc (Spelter) (B 6 - 58).

Tentative Specifications for:

Nickel Seamless Pipe and Tube (B 161 - 58 T),
Nickel-Copper Alloy Seamless Pipe and Tube (B 165 - 58 T), and
Nickel-Chromium-Iron Alloy Seamless Pipe and Tube (B 167 - 58 T).

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Specification for Remelted Lithium Metal in Ingot Form as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for Solder Metal (B 32 - 58 T):[2]

Section 1(a).—Revise the first sentence to read: "These specifications cover 32 grades of tin-lead, tin-lead-antimony, tin-antimony, and silver-lead alloys in any form, formerly known as soft solder."

Table I.—Revise to read as shown in the accompanying Table I.[3] New grades 63B and 95TA are added, minimum antimony requirements have been added for the "B" grades, copper is specified as 0.08 max for all grades, and arsenic limits have been added.

Section 4.—To the Note add the following sentence: "Alloy grade 95TA designates a composition of 95 tin–5 antimony."

Section 5(d).—Add the words "except for alloy grade 95TA."

Table II.—Add information on new alloy 63B with nominal composition of 63 tin, 37 lead as follows:

Specific gravity	8.40
Solidus:	
deg Cent	183
deg Fahr	361
Liquidus:	
deg Cent	183
deg Fahr	361
Uses	For soldering of printed electrical circuits and as "Fine Solder."

Add a new section to Table II titled "Tin-Antimony Alloy" including information on new alloy 95TA with nominal composition of 95 tin, 5 antimony as follows:

Specific gravity	7.25
Solidus:	
deg Cent	234
deg Fahr	452
Liquidus:	
deg Cent	240
deg Fahr	464
Uses	For electrical and joints on copper for plumbing and heating.

---

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

[2] 1958 Book of ASTM Standards, Part 2.

[3] The revised table is not included in this report but appears in the revised specification, see 1960 Supplement to Book of ASTM Standards, Part 2.

Change footnote *a* of Table II to read: "Federal specifications similar to and also those for ASTM alloy grades 70B, 63B, 60B, 50B, and 40B contain antimony in the range of 0.20 to 0.50 per cent in addition to a permissible limit of 0.25 per cent bismuth. Such formulation is intended to provide for reliability. . . ."

Tentative Specification for Rosin Flux Cored Solder (B 284 – 58 T):[2]

Section 1.—Revise to read as follows:

1. These specifications cover cored solder containing specified weight percentages of natural, modified, activated rosin or ductile form of the same, as specified.[3]

Section 2.—Change the first sentence of the Note by the addition of "(NR)" following the words "2.2 per cent natural rosin flux."

Section 6.—Change to read as follows:

6. *Types of Flux.*—The flux making up the core or cores of the solder shall be one of the following types (see Table III):

Natural Rosin (*NR*).—Gum or wood rosin at least equivalent to grade WW[5] of the Methods of Sampling and Grading Rosin (ASTM Designation: D 509), and in the solid or powder form. Additive agents are prohibited. This flux is the least corrosive of the known soldering fluxes.

Modified Rosin (*MR*).—Rosin conforming to that defined as Rosin Modified in the Definitions of Terms Relating to Naval Stores and Related Products (ASTM Designation: D 804) and in solid or powder form. These rosins have been chemically or thermally treated. Some of these are no more corrosive than natural rosin.

Ductile Rosin (*D*) (formerly known as "Plastic Rosin").—Rosin, specified as NR or MR, to which is added gum spirits of turpentine in accordance with the Specifications for Spirits of Turpentine (ASTM Designation: D 13) for the purpose of facilitating manufacturing and soldering operations. Diluents, other than turpentine, may be provided under this specification.[6] Diluent and its weight percentage shall be as agreed upon by the manufacturer and purchaser.

Activated Rosin (*A*).—Rosin, specified as NR or MR, to which is added an activating agent to facilitate soldering. Upon request, the manufacturer shall furnish a certified statement that the composition or performance, or both, shall be as specified by mutual agreement between the manufacturer and the purchaser. This flux shall be free from any materials which might have a toxic effect on persons using these solders in production soldering.

Other combinations of grades, which shall be as agreed upon by the manufacturer and the purchaser, may be provided under these specifications.

New Table.—Add a new Table III to read as shown in the accompanying Table II, renumbering subsequent tables accordingly.

TABLE II.—SPECIFIED TYPES OF ROSIN FLUX CORES.
(New Table III, Specification B 284)

Grade	Description
NR	Natural rosin
MR	Modified rosin
DNR	Ductile natural rosin
DMR	Ductile modified rosin
DNRA	Ductile natural rosin, activated
DMRA	Ductile modified rosin, activated

Table III.—Renumber as Table IV and in the last line change the number of samples for a lot size of over 10,000 lb from the present "10" to read "10 or more.[a]" The footnote *a* will read "[a]Add 10 samples for each increment of 10,000 lb in lot size."

Section 9.—Revise the first sentence of Paragraph (*a*) to read as follows: "Select a minimum of 20 g of cored solder from each of the 6-ft lengths and wipe the surface with a clean cloth."

At the end of Paragraph (*b*) add the sentence: "The average weight per cent of flux reported should be derived from at least three samples."

---

[3] Valuable information on the use of solders and fluxes may be found in ASTM Special Technical Publication *STP No. 189*, "Symposium on Solder," June, 1956.

[5] The purpose of specifying water white rosin WW is to facilitate inspection for quality of soldering by visual observation through the rosin flux residue on the finished joint.

[6] Where soldering is done in electrical equipment enclosing operating contacts, certain vapors from the flux tend to cause excessive pitting of the contacts.

Appendix.—Delete the Appendix if the above revisions are approved.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specification for Fire-Refined Casting Copper (**B 72 - 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives since they are either in process of revision or are being reviewed by the responsible subcommittee:

Tentative Specifications for:

Nickel-Copper Alloy Plate, Sheet, and Strip (**B 127 - 58 T**),
Nickel Rod and Bar (**B 160 - 58 T**),
Nickel Seamless Pipe and Tube (**B 161 - 58 T**),
Nickel Plate, Sheet, and Strip (**B 162 - 58 T**),
Seamless Nickel and High Nickel Alloy Condenser and Heat Exchanger Tubes (**B 163 - 58 T**),
Nickel-Copper Alloy Rod and Bar (**B 164 - 58 T**),
Nickel-Copper Alloy Seamless Pipe and Tube (**B 165 - 58 T**),
Nickel-Chromium-Iron Alloy Rod and Bar (**B 166 - 58 T**),
Nickel-Chromium-Iron Alloy Seamless Pipe and Tube (**B 167 - 58 T**),
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (**B 168 - 58 T**),
Titanium and Titanium Alloy Strip, Sheet, and Plate (**B 265 - 58 T**),
Titanium Sponge (**B 299 - 58 T**),
Refined Secondary Lead (**B 325 - 58 T**),
Nickel-Molybdenum and Nickel-Molybdenum-Chromium Alloy Castings (**B 332 - 58 T**),
Nickel-Molybdenum Alloy Plate and Sheet (**B 333 - 58 T**),
Nickel-Molybdenum-Chromium Alloy Plate and Sheet (**B 334 - 58 T**),
Nickel-Molybdenum Alloy Rod (**B 335 - 58 T**),
Nickel-Molybdenum-Chromium Alloy Rod (**B 336 - 58 T**),
Seamless and Welded Unalloyed Titanium Pipe (**B 337 - 58 T**), and
Seamless and Welded Unalloyed Titanium Tubing (**B 338 - 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (W. E. Milligan, chairman) recommended that Specification B 72 be advanced to standard.

The Canadian Standards Assn. has adopted Specifications B 115 and B 224 and a note to this effect will be included on the specifications.

Subcommittee II on Lead (Sidney Rolle, chairman).—The Task Group on Pig Lead will meet before the Annual Meeting to discuss Specification B 29 with a view to revising the scope clause. Data are being collected for the writing of a specification on nuclear lead or lead shielding.

Subcommittee III on Tin Base and Lead Base Alloys (G. H. Clamer, chairman).—Specification B 23 (Babbitt Metal) was revised and reverted to tentative by the ASTM Administrative Committee on Standards November 6, 1959. Negative ballots on revisions of Specifications B 32 (Solder Metal) and B 284 (Rosin Flux Solder) have been resolved and the proposed revisions are included in this report.

Progress has been made on the basic planning for the 1961 Symposium on Solders of which P. R. White, Bell Laboratories, is chairman.

Subcommittee IV on Refined Zinc and Wrought Zinc (D. W. Pettigrew, chairman).—A task group formed to prepare a specification on cast and wrought zinc anodes for cathodic protection met in Chicago, Feb. 3, and a report on the specification will be submitted at the June 1960 meeting.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VII on Refined Nickel and Cobalt, High Nickel Alloys and High Cobalt Alloys, Cast and Wrought (V. W. J. Franceschini, chairman).—A ballot will be circulated to the subcommittee members recommending that nickel-base alloy specifications which have stood for two years be advanced to standard.

A specification covering wrought nickel and nickel alloy welding fittings was prepared by a task group and is being circulated for subcommittee approval.

Where required, B-2 specifications covering nickel and nickel alloys will be changed to conform with heat treating terms in Specification E 44.

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman).—Meetings were held throughout the year by the task groups set up to study the less common metals, and a number of specifications have been drafted, as follows: A specification covering molybdenum sheet metal is in final draft and four other specifications are in progress. Specifications are being formulated by the Tungsten Task Group. A tentative specification on primary columbium metal has been completed. The Tantalum Task Group discussed a specification for unalloyed tantalum but no action has been taken on the specification. The tentative specification for lithium appended to this report was prepared by the Lithium Task Group. Specifications covering zirconium sponge; tubes; ingot; bar, rod and wire; sheet, strip and plate, for nuclear applications, were approved by Committee B-2 and the Administrative Committee on Standards. A specification covering zirconium alloy forgings and extrusions for nuclear applications is being balloted by the subcommittee. A Sodium Task Group has been organized. A rough draft of a specification covering thorium has been prepared by the Thorium Task Group. No action has been taken by the Uranium Task Group. A specification covering beryllium is being held in abeyance until agreement is reached on basic composition problems.

Subcommittee IX on Titanium and Titanium Alloys (A. F. Jones, chairman) prepared the new Specification for Titanium and Titanium Alloy Bars and Billets (B 348), referred to above under actions by Administrative Committee on Standards. A specification for titanium and titanium alloy castings is under discussion, and a specification for seamless and welded unalloyed titanium welding fittings has been prepared and will be circulated for approval. A specification will be prepared covering titanium forgings. A specification was prepared for titanium and titanium alloy welding rod and bare electrodes and submitted to the Joint AWS-ASTM Committee on Filler Metal for action.

Task Groups are presently working on standard nomenclature; flare testing of titanium tubing; and stress and strain versus temperature data for use in connection with unfired pressure vessels (ASME Case No. 1258).

Action will be taken by the subcommittee regarding the advancement of Specification B 299 to standard. It is expected that recommendation will be made to retain Specifications B 265, B 337, and B 338 as tentative.

This report has been submitted to letter ballot of the committee which consists of 146 members, 100 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

A. M. BOUNDS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Zirconium and Zirconium Alloy Forgings and Extrusions for Nuclear Applications (B 356 – 60 T).

Revision of Tentative Specifications for:

Solder Metal (B 32 – 60 T).

The new Tentative Specifications B 356 – 60 T were accepted by the Standards Committee on September 29, 1960 and they appear in the 1960 Supplement to Book of ASTM Standards, Part 2. The revision of Tentative Specifications B 32 – 60 T was accepted on December 20, 1960, and the revised specifications are available as a separate reprint.

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held two meetings during the past year: in Atlantic City, N.J., on June 22, 1959, and in Chicago, Ill., on Feb. 1, 1960.

The committee consists of 95 members, of whom 80 are voting members; 32 are classified as producers, 26 as consumers, and 22 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, K. G. Compton.
Vice-Chairman, E. K. Camp.
Secretary, W. H. Ailor.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Total Immersion Corrosion Test of Non-Ferrous Metals (**B 185 - 43 T**),[1] and
Alternate Immersion Corrosion Test of Non-Ferrous Metals (**B 192 - 44 T**).[1]

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives:

Tentative Method of Salt Spray (Fog) Testing (B 117 - 57 T):[2]

Section 6.—Revise the last sentence to read as follows: "The pH measurement shall be made electrometrically at 25 C (77 F) using a glass electrode with a saturated potassium chloride bridge; or colorimetrically using bromthymol blue as indicator (Note 4)."

Tentative Method of Acetic Acid - Salt Spray (Fog) Testing (B 287 - 57 T):[2]

Section 6.—Change the third sentence to read as follows: "The pH of this solution shall be adjusted to the range 3.1 to 3.3 by the addition of less than 1 per cent of acetic acid (Notes 2 and 3)." Add to the next to last sentence the words "at 25 C (77 F)."

New Note.—Add a new Note 3 to Section 6, to read as follows:

Note 3.—If less than 0.2 or more than 0.3 per cent of glacial acetic acid is required to attain the specified pH, the purity of the water and/or the salt may not be satisfactory.

Section 8(b).—Delete the last sentence.

The above changes are required to clarify the method for controlling the acetic acid concentration and pH and to eliminate conflict between Sections 6 and 8.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Test (C. O. Durbin, chairman).—A task force is continuing its studies on the effect of grades of salts on the salt spray test. The work to date indicates that impurities found in the usual grades of salt have little effect.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 3.

[2] 1958 Book of ASTM Standards, Parts 3 and 8.

The subcommittee has drafted a method for the copper-accelerated salt spray test (Cass test) and plans to prepare a method for the copper-accelerated clay-pack humidity test (Corrodekote test).

Subcommittee VI on Atmospheric Corrosion (H. R. Copson, chairman).—In connection with the 1957 Test Program,[3] the 1-yr aluminum specimens were removed during the fall of 1959 and tests are currently under way on these specimens. It is anticipated that the results will be incorporated into a report which will probably be appended to the 1961 Annual Report of Committee B-3.

Subcommittee VII on Weather (C. P. Larrabee, chairman).—Another program of calibrating the corrosivity of atmospheric test sites was started about May 1, 1960. In this program, there are 24 test sites in the United States, 8 in Canada, 4 in Panama, 2 in England, and 1 in the Philippines. The subcommittee sponsored a paper by P. J. Sereda, National Research Council of Canada, on "Measurement of Surface Moisture and Sulfur Dioxide Activity at Corrosion Sites" for publication in the ASTM BULLETIN.[4] Apparatus developed by Mr. Sereda will be used at 4 sites in the United States and at 4 sites in Canada to study corrosivity of atmospheres as measured by sulfur dioxide content, length of time specimens are wet, and temperature of the specimens. These results will be compared to monthly losses of specimens of steel, zinc, and aluminum.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (G. V. Kingsley, chairman).—The examination and evaluation of the 15-yr exposures of stainless steel galvanic couples (1941 program) is nearly completed. The first set of samples from the magnesium panel type galvanic test program (1958) has been brought in and dismantling and cleaning of the panels has been started in preparation for evaluation by measurements of mechanical properties.

During the past year, a task group has been active in studying the problem of corrosion by high-temperature boiler feedwater. This work is continuing, and it is hoped that during the coming year this task group will be ready to define the problem and recommend a course of subcommittee action.

Another task group has just been appointed to investigate the problem of corrosion of aluminum conductors and accessory hardware and to evaluate the interest in and need for corrosion studies in this field.

This report has been submitted to letter ballot of the committee, which consists of 80 voting members; 61 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

A. W. TRACY,
Secretary.

---

[3] The details of this program were outlined in the Report of Subcommittee VI on Atmospheric Corrosion: 1957 Test Program, which appears as Appendix I to the 1959 Annual Report of Committee B-3 in the 1959 *Proceedings*, p. 176.

[4] ASTM BULLETIN, No. 246, May, 1960, p. 47 (TP 107).

REPORT OF COMMITTEE B-4

ON

METALLIC MATERIALS FOR THERMOSTATS AND FOR ELECTRICAL RESISTANCE, HEATING AND CONTACTS*

Committee B-4 on Metallic Materials for Thermostats and for Electrical Resistance, Heating and Contacts held three meetings during the year: in Atlantic City, N. J., on June 26, 1959; in Pittsburgh, Pa., on Oct. 30, 1959; and in Washington, D. C., on Feb. 24, 1960.

The committee consists of 75 members, of whom 26 are classified as producers, 34 as consumers, and 15 as general interest members.

During the past year, with approval of the Board of Directors, Committee B-4 has changed its title to that which appears above. The new title includes thermostat metals which have long been included in the committee's scope.

As it has done in the past, Committee B-4 again cooperated with The Pennsylvania State University in an Engineering Seminar on Electrical Contacts. The Seminar was held from June 5 to 10, 1960. The committee continues to sponsor the Bibliography and Abstracts on Electrical Contacts published as *ASTM STP 56G* and annual supplements. In 1959, the Society published Supplement *ASTM STP 56M* covering the literature and abstracts for 1958.

During the past year, Subcommittee IV on Contact Materials has had a special speaker at each of its meetings. At the Annual Meeting in Atlantic City, Frank Anders of the U. S. Navy Bureau of Ships discussed "Thermoelectric Materials." At the Pittsburgh meeting S. W. Chaikin of Stanford Research Institute discussed "Chemical Investigation of Contact Contamination in Sealed Relays." At the February meeting Erle I. Shobert II of Stackpole Carbon Co. presented a paper on "Simulated Altitude Carbon Brush Investigations."

At the February meeting in Washington, George F. Geiger was elected an Honorary Member of Committee B-4 for his outstanding services and activities in Subcommittee II. The other Honorary Members of this committee are P. H. Brace, F. E. Carter, Ragnar Holm, and R. D. Van Nordstrand.

At the 1959 Annual Meeting, C. K. Strobel, Secretary, received the Society's Award of Merit for his outstanding work as secretary of Subcommittee IV of Committee B-4 since 1946 and as secretary of Committee B-4 since 1956.

The committee was grieved to learn of the death of C. S. Brainin. Mr. Brainin had been active in the work of Subcommittee IV.

The officers elected for the ensuing term of two years are as follows:

Chairman, E. I. Shobert II.
Vice-Chairman, Leonard Mayeron.
Secretary, C. K. Strobel.
Assistant Secretary, C. L. Guettel.

Revision of Tentative

The committee recommends revisions as appended hereto[1] of the Tentative Specifications for High Resistivity, Low-

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

Temperature-Coefficient Wire (**B 267 – 53 T**),[2] and continuation of the specifications as tentative.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Recommended Practice for Dimensional Standards for Projection Welding Contacts (**B 321 – 57 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of the Standard Specifications for Drawn or Rolled Nickel-Chromium and Nickel-Chromium-Iron Alloys for Electrical Heating Elements (**B 344 – 59**):[3]

Section 11 (*a*).—In the table on Spooling Weights, delete the size ranges "Up to 0.00175 in." and "0.00175 to 0.0035 in., excl."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Electrical Heating and Resistance Materials (C. W. Armstrong, chairman), submitted the proposed revisions of Specifications B 267 mentioned earlier in the report. The revisions are extensive, including both refinement of many requirements and addition of much new material, and have occupied the major portion of the subcommittee's activities during the year.

Method B 84 has been studied by a task group, and a proposed revision is nearly ready. In addition, the subcommittee is considering whether a specification covering iron-chromium-aluminum alloys for electrical heating applications can be developed at this time; has established a better liaison with military specification-writing agencies, and with Subcommittee X on Magnet Wire Insulation of Committee D-9 on Electrical Insulating Materials on film-type insulations for wire.

Subcommittee II on Wrought and Cast Alloys for Structural Use at High Temperatures continues to be inactive.

Subcommittee III on Thermostat Metals (Unto Savolainen, chairman) has carried out a successful membership campaign adding about 10 new members, particularly in the consumer group, during the year. A new test method entitled "Mechanical Torque Rate of Spiral Coils of Thermostat Metal" is being completed. Subjects under consideration by the enlarged subcommittee include: (1) extension of the range of the flexivity test (B 106) to include material thinner than 0.015 in.; (2) thermal emissivity test methods; (3) thermal conductivity test methods; (4) corrosion tests; (5) compilation of a bibliography on the subject of thermostat metals; (6) a test method for the thermal rotation of spiral coils of thermostat metal; (7) a test for uniformity of thermostat metal when subjected to mechanical deformation; and (8) a bond strength test.

Subcommittee III has recommended that the Standard Method of Test for Linear Expansion of Metals (B 95 – 39) be transferred from the jurisdiction of Committee B-4 to that of Committee E-1 on Methods of Testing. This transfer will take place when the recommendation for transfer is accepted by Committee E-1.

Subcommittee IV on Contact Materials

---

[2] 1958 Book of ASTM Standards, Part 2.

[3] 1959 Supplement to Book of ASTM Standards, Part 2.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

(J. D. Kleis, chairman).—Extensive work during the year was directed toward revising and broadening method B 182. The work on physical properties was expanded, with most of the effort directed toward development of a simplified device for determining thermal conductivity of contact materials. A continuation of the work of listing and defining terms used in the contact field was sponsored by the Standardization Section, with substantial progress shown. The Section on High Current Contacts has gathered considerable data on arc erosion of the common contact materials, using as the unit of measure the erosion in volume per coulomb.

Microcontacts are being investigated for operation at 0.1 g operating force. It has been found that contact cleaning plays a very important role, and a considerable amount of work is being devoted to this portion of the problem.

Interest in static connectors and connections has been shown by the substantial progress made in this area.

Test racks for holding a number of contact fixtures have been designed. Arrangements have been made to conduct tests on several contact materials in industrial, marine, and rural atmospheres at ASTM test sites.

The newest section, devoted to the study of sliding contacts, has made substantial progress. Some test devices have been constructed, and others are under construction. Tests are planned to provide information toward development of standard tests.

This report has been submitted to letter ballot of the committee, which consists of 76 members; 42 members have returned their ballots, of whom 37 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ERLE I. SHOBERT II,
Chairman.

C. K. STROBEL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards the recommendation that a Method of Test for Mechanical Torque Rate of Spiral Coils of Thermostat Metal be published as tentative. This recommendation was accepted by the Standards Committee on September 29, 1960, and the new tentative method appears in the 1960 Supplement to Book of ASTM Standards, Part 3, bearing the designation B 362 - 60 T.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys held two meetings during the year: at Washington, D. C., in September 1959 and at Philadelphia, Pa., in February 1960.

The committee consists of 130 members; 45 are classified as consumers, 58 as producers, 21 as general interest members, 5 as producers and consumers, and 1 as a consultant.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. H. Jennings.

First Vice-Chairman, W. D. France.

Second Vice-Chairman, William Romanoff.

Secretary, Gordon C. Mutch.

The Supervisory Group of the committee continued to function as advisor to the American Standards Assn. holding the secretariat of Technical Committee 26 on Copper and Copper Alloys of the International Organization for Standardization (ISO). Not having obtained wide enough support from industry, the Supervisory Group to ASA on ISO/TC-26 recommended that the USA relinquish the Secretariat, but continue as a participating member.

Action has been taken on the following items:

Draft ISO Recommendation No. 310—Method of Test for Expansion of Copper and Copper Alloy Tubes

Draft ISO Recommendation No. 311—Method of Mercurous Nitrate Test for Copper and Copper Alloys

Draft ISO Recommendation No. 312—Classification of Coppers

The four working groups in ISO/TC 26, with the respective United States members, are as follows: WG1, Mechanical Testing, B. L. Wilson; WG2, Classification of Wrought Copper and Copper Alloys, A. E. Moredock; WG3, Classification of Cast Copper and Copper Alloys, J. S. Roberts; and WG4, Classification of Coppers, A. E. Beck. WG4 was established at the 1958 meeting of ISO/TC 26 at Harrogate, England, with Mr. Beck as chairman.

During the Washington meetings a paper entitled "The Magnetic Properties of Copper and Copper Alloys," by F. C. Parker, R. M. Winter, and J. A. LaPorta,[1] was presented.[2]

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending 3 new specifications for publication—one as tentative, and 2 as standards for immediate adoption; revision of 2 tentatives; adoption of 1 tentative as standard with revision; and revision of 19 standards for immediate adoption.

The standards and tentatives affected, together with the revisions recommended, are covered in detail in the Appendix to this report.[3]

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Published in *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 230 (1959).

[2] A paper by G. R. Gohn and W. G. Bader, "The Tensile Properties of Hard-Drawn Alloy A Phosphor Bronze Rod," presented Sept. 14, 1960, during the meeting of Committee B-5 at Niagara Falls, N. Y., appears on p. 843 of this volume.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee W-1 on Plate, Sheet and Strip (A. E. Moredock, chairman) is recommending the adoption of Specification B 291 as standard. The methods of conducting the embrittlement test in Specification B 152 were modified to reflect a maximum limit on the magnification used for the examination of the test specimen. At high magnification, disputable items become apparent which are not a factor in the tests. Specifications B 36, B 103, B 121, B 122, B 152, and B 291 were revised to cover consumer needs for standard tempers as well as special or non-standard tempers. Data on copper foil for printed circuit quality are being compiled to be included in a future specification.

Subcommittee W-2 on Rod, Bars, and Shapes (O. A. Klema, chairman) has revised Specification B 16 to expand the size range of hard temper material currently available within the industry, and is recommending revisions in Specifications B 133, B 187, and B 249. Task groups are considering revisions in Specifications B 16, B 21, B 140, B 151, B 196, and B 283. A task group (joint with Subcommittee W-4) is studying the problem of surface oxidation of oxygen-free copper rod. Task groups are preparing new specifications for leaded brass rod, copper-zinc-silicon rod, and copper-nickel-silicon rod and bar.

Subcommittee W-3 on Wire and Wire Rod (M. J. Mianulli, chairman) is recommending that Paragraph 1(*b*) be deleted from Specification B 250 in order to eliminate overlapping with Specification B 249. The magnification for microscopic examination for embrittlement (Paragraph 8) of Specification B 272 was changed to read "from 75 to 200 diameters, inclusive." Study of the scope in Specification B 159 revealed that it need not be expanded to include rectangular and square wire as little usage and interest was reported. Tasks groups are studying (1) the addition of mechanical properties table for rolled flat wire to Specification B 134; (2) minimum size of wire to which straightness tolerances would apply in Specification B 250; (3) terms relating to heat treatment in Specification B 197; and (4) tensile requirements for annealed temper of alloy B in Specification B 99. A new specification for a copper-nickel-silicon alloy wire is being developed.

Subcommittee W-4 on Pipe and Tube (G. C. Mutch, chairman) is recommending the adoption as standard of the tentative revision of a portion of Specification B 251 now appearing as an editorial note. The magnification requirement for microscopic examination for embrittlement has been changed to read "from 75 to 200 diameters, inclusive" in Specifications B 68, B 75, B 188, and B 280. Revisions have been made in Table III of Specification B 280. Modification of length requirements in Specification B 302 were approved. The requirement for microscopic examination of DHP copper in Specification B 280 has been eliminated. Dimensional tolerances for square and rectangular tube for electronic uses have been added to Specifications B 75, B 135, B 188, and B 251. Specification B 135 has been expanded to include 90-10 commercial bronze alloy. The sampling and retest provisions of Specifications B 188 and B 251 have been clarified. Two new specifications, for finned heat exchanger tube and copper capillary tube, have been approved to reflect special industrial usage. Revisions were made in Table I of Specification B 111 and requirements for drawn and stress relieved 70-30 copper nickel were added.

---

[3] See p. 149.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Items under study are specifications for surface oxidation of oxygen-free copper tube, rectangular waveguide tube, "U" bent copper-nickel heat exchanger tube, and residual stresses in condenser tube, addition of alloy D to Specification B 315, and the establishment of Specification B 88 as a separate product specification. A revision of Specification B 251 is being prepared by the subcommittee.

Subcommittee F-1 on Castings and Ingots for Remelting (G. H. Clamer, chairman).—A Specification Review Task Group has been appointed to revise all specifications under the subcommittee's jurisdiction in order to bring all specifications to a uniform ASTM approved form and to correct minor discrepancies in chemical and physical requirements. Specifications B 61 and B 62 have been rewritten. A specification has been written for copper-nickel block, ingot, and shot to cover an additive for copper-base alloys. Specifications for investment castings and copper-nickel alloy castings are being developed. The permissibility of impregnation of copper-base alloy castings is being studied. Several military specification study programs were carried out by the members of Subcommittee F-1.

Subcommittee G-1 on Methods of Test (P. D. Strubhar, chairman) has submitted recommendations on the use of hardness correction on cylindrical surfaces to Committee E-1 on Methods of Testing. A number of ISO/TC 26 items on tests relating to copper-base alloy products are being studied. The ISO/TC 26 (53E) recommendation on the Pin Test was voted as compatible with Specification B 143; the ISO/TC 26/WG1 recommendation on Mechanical Tests is held in abeyance until the revised draft is received.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman).—Appropriate dimensional tolerances for square and rectangular tubes were recommended to Subcommittee W-4. Tolerances on minimum size of square and rectangular wire are being generated for Subcommittee W-3.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) is currently making a review of the format of the tables in Committee B-5 specifications. Revisions have been proposed for the sampling and retesting requirements in Specifications B 155, B 188, and B 251. Similar revisions will be made in other standards.

This report has been submitted to letter ballot of the committee, which consists of 130 members; 78 members returned their ballots, of whom 75 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. JENNINGS,
Chairman.

L. H. ADAM,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Specification for Bronze Castings for Bridges and Turntables (B 22 – 52) be revised and reverted to tentative. This recommendation was accepted by the Standards Committee on December 5, 1960, and the revised tentative specification is available as a separate reprint.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix, recommendations are given affecting certain specifications, both standard and tentative, referred to earlier in the report. The specifications appear in their present form in the 1958 Book of ASTM Standards, Part 2, or in the 1959 Supplement to Book of ASTM Standards, Part 2.

NEW STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the specifications as appended hereto[1] be approved for reference to letter ballot of the Society for adoption as standard, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the specifications may be referred to letter ballot of the Society:

Standard Specifications for:

Copper and Copper-Alloy Seamless Condenser and Heat Exchanger Tubes with Integral Fins, and

Hard-Drawn Copper Capillary Tube for Restriction Applications.

NEW TENTATIVE

The committee recommends for publication as tentative the Specifications for Copper-Nickel Alloys in Ingot, Slab, or Shot Form as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for General Requirements for Wrought Copper-Alloy Wire (B 250 – 58a T):

Section 1(b).—Delete this paragraph.

Tentative Specification for Flat Copper Products with Finished (Rolled or Drawn) Edges (Flat Wire and Strip) (B 272 – 58 T):

Section 8.—Change the magnification requirements from the present "not less than 75 diameters" to read "from 75 to 200 diameters, inclusive."

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specification for Copper-Zinc-Manganese Alloy (Manganese Brass) Sheet and Strip (**B 291 – 58 T**) be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Section 1(b).—Delete this paragraph.

Section 6.—Change to read as follows:

6. The standard tempers of rolled material are as designated in Table I in the column entitled "Rolled Temper." Special or nonstandard tempers are subject to negotiation between manufacturer and purchaser (Section 2 (*l*)).

---

[1] The new standards and tentative appear in the 1960 Supplement to Book of ASTM Standards, Part 2.

Section 7.—Change to read as follows:

7. The standard tempers of annealed material are as designated in Table II in the column entitled "Nominal Grain Size." Special or nonstandard tempers are subject to negotiation between manufacturer and purchaser (Section 2 (*1*)).

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Free-Cutting Brass Rod, Bar, and Shapes for Use in Screw Machines (B 16 – 58):

Table I.—Change the values for Hard Temper Rounds, Hexagons and Octagons to read as follows:

Diameter or Distance Between Parallel Surfaces, in.	Tensile Strength, min, psi	Yield Strength, min, psi	Elongation in 4 × Diameter or Thickness of Specimen, min, per cent
$\frac{1}{16}$ to $\frac{3}{16}$ incl.	80 000	45 000	...
Over $\frac{3}{16}$ to $\frac{1}{2}$ incl.	70 000	35 000	4
Over $\frac{1}{2}$ to $\frac{3}{4}$ incl.	65 000	30 000	6

Standard Specification for Brass Plate, Sheet, Strip, and Rolled Bar (B 36 – 56):

Section 1(b).—Delete this paragraph.

Section 5.—Change to read as follows:

5. The standard tempers of rolled material are as designated in Table II in the column entitled "Rolled Temper." Special or non-standard tempers are subject to negotiation between manufacturer and purchaser (Section 2 (*5*))."

Section 6.—Change to read as follows:

6. The standard tempers of annealed material are as designated in Table III in the column entitled "Nominal Grain Size." Special or nonstandard tempers are subject to negotiation between manufacturer and purchaser (Section 2 (*5*)).

Standard Specification for Steam or Valve Bronze Castings (B 61 – 52):

Revise as appended hereto.[2]

Standard Specification for Composition Brass or Ounce Metal Castings (B 62 – 52): Revise as appended hereto.[2]

Standard Specification for Seamless Copper Tube, Bright Annealed (B 68 – 58):

Section 9.—Change the magnification requirements from the present "not less than 75 diameters" to read "from 75 to 200 diameters inclusive."

Standard Specification for Seamless Copper Tube (B 75 – 59):

Section 1.—Change the first sentence to read: "This specification covers seamless copper, round and rectangular including square tube suitable for general engineering purposes."

Section 2.—Renumber as Section 3.

Section 3.—Renumber as Section 2, and change the title from "Basis of Purchase" to "Ordering Information." In Item (*4*) insert the words "or distance between parallel surfaces" after the word "Diameter."

Section 5.—Add the following sentences: "Rectangular including square tubes shall normally be supplied only in drawn (general purpose) temper. When there is a need for light-drawn or hard-drawn tempers these may be supplied as agreed upon between the manufacturer and the purchaser."

Table II.—Change the first column heading to read "Temper," and change the temper designations "Light" and "Hard" to read "Light-Drawn[b]" and

---

[2] The revised standards appear in the 1960 Supplement to Book of ASTM Standards, Part 2.

"Hard-Drawn[b]." Change the second column heading to read: "Outside Diameter or Major Distance Between Outside Parallel Surfaces, in."

Change the first sentence of footnote *a* to read: "Rockwell hardness values shall apply only to tubes having a wall thickness of 0.020 in. or over and to round tubes having an inside diameter of $\frac{5}{16}$ in. or over and to rectangular including square tubes having an inside major distance between parallel surfaces of $\frac{3}{16}$ in. or over."

Add a new footnote *b* to read: "[b]Light-drawn and hard-drawn tempers are normally available in round tube only."

Table III.—In the first column change the temper designation from "Soft" and "Light" to read: "Soft anneal" and "Light anneal." Change the second column heading to read "Outside Diameter or Major Distance Between Outside Parallel Surfaces, in."

Change the first sentence of footnote *a* to read: "Rockwell hardness values shall apply only to tubes having a wall thickness of 0.015 in. or over and to round tubes having an inside diameter of $\frac{5}{16}$ in. or over, and to rectangular including square tubes having an inside major distance between parallel surfaces of $\frac{3}{16}$ in. or over."

Section 7.—Change the second sentence to read: "Tension tests are required for tubes with a wall thickness under 0.020 in. and for round tubes having an inside diameter under $\frac{5}{16}$ in., and for rectangular including square tubes having a major distance between inside parallel surfaces under $\frac{3}{16}$ in."

Section 9.—Change the title to read: "Expansion Test for Round Tube."

Section 10.—Change the title to read: "Hydrostatic Test for Round Tube."

Section 13.—Change the magnification requirements from "not less than 75 diameters" to read "from 75 to 200 diameters, inclusive."

Section 14.—Change Paragraphs (*b*), (*c*), and (*g*) and add a new Paragraph (*h*) to read as follows:

(*b*) *Wall Thickness Tolerances.*—See Section 8(*d*), Tables V and XI.

(*c*) *Tolerances for Diameter or Distance Between Parallel Surfaces.*—See Section 8(*e*), Tables VI and XII.

(*g*) *Straightness Tolerances.*—For round tubes see Section 8(*j*)(*1*), Table X. For rectangular including square tubes see Section 8(*j*)(*2*).

(*h*) *Corner Radius for Rectangular Including Square Tubes.*—See Section 8(*k*), Table XIII.

Tables IV and V.—In each table change the heading of the first column to read: "Outside Diameter or Major Distance Between Parallel Outside Surfaces, in."

Table VI.—Change the title to read: "Schedule of Mill Lengths with Ends, in Coils." Change the heading of the first column to read as shown above for Tables IV and V.

Standard Specification for Phosphor Bronze Plate, Sheet, Strip, and Rolled Bar (B 103 – 55):

Section 1(b).—Delete this paragraph.

Section 6.—Change to read as follows:

6. The standard tempers of rolled material are as designated in Table II in the column entitled "Temper." Special or non-standard tempers are subject to negotiation between manufacturer and purchaser (Section 3 (*2*)).

Standard Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 58):

Section 4.—In Paragraph (*a*) delete the words "70-30 copper-nickel." Add a new Paragraph (b) to read as follows, relettering the present Paragraph (*b*) and subsequent paragraphs accordingly:

(*b*) 70-30 copper-nickel shall be supplied in one of the following tempers as specified:

Annealed
Drawn, stress-relieved

Table I.—Delete aluminum brass alloys types C and D, and 80–20 copper-nickel alloy type B. For 80–20 copper-nickel tube, type A, change the iron requirement from the present "0.6 max" to read "0.5 to 1.0." Change footnote *c* to read: "Types A, C, and D aluminum brasses and Type B 80–20 copper-nickel have been discontinued."

Section 8 (a).—Change to read as follows:

8. (*a*) The tubes selected for test shall be capable of withstanding the following expansion of its inside diameter at one end by driving a pin having a taper of 1½ in. per ft into the tube. The expanded tube shall show no cracking or rupture visible to the unaided eye.

Temper	Alloy	Expansion of Tube Inside Diameter, in per cent of Original Inside Diameter
Annealed	Muntz metal	15
	Admiralty	20
	Red brass	20
	Aluminum brass	20
	Aluminum bronze	20
	70-30 copper-nickel	30
	80-20 copper-nickel	30
	90-10 copper-nickel	30
Light-drawn	Copper	20
	Arsenical copper	20
	90-10 copper-nickel	30
Drawn, stress-relieved	70-30 copper-nickel	20

Table II.—Add a fifth column to the table, headed "Elongation in 2 in., min, per cent."

Below the line for 70-30 copper-nickel, annealed, add a new line for 70-30 copper-nickel, drawn, stress-relieved, with the following values:

Material	70-30 copper-nickel
Temper	Drawn, stress-relieved
Tensile strength, min, psi	72 000
Yield strength, min, psi	50 000
Elongation in 2 in., min, per cent:	
For wall thicknesses up to 0.048 in., incl	12
For wall thicknesses over 0.048 in., incl	15

Standard Specification for Leaded Brass Plate, Sheet, Strip, and Rolled Bar (B 121 – 55):

Section 1(b).—Delete this paragraph.

Section 5.—Change to read as follows:

5. The standard tempers of rolled material are as designated in Table II in the column entitled "Temper." Special or non-standard tempers are subject to negotiation between manufacturer and purchaser (Section 3 (*2*)).

Section 6.—Change to read as follows:

6. The standard tempers of annealed material are as designated in Table IV in the column entitled "Temper, Nominal Grain Size." Special or nonstandard tempers are subject to negotiation between manufacturer and purchaser (Section 3 (*2*)).

Standard Specification for Copper-Nickel-Zinc Alloy (Nickel Silver) and Copper-Nickel Alloy Plate, Sheet, Strip, and Rolled Bar (B 122 – 59):

Section 1(b).—Delete this paragraph.

Section 5.—Change to read as shown above for Section 6 of Specification B 103.

Section 6.—Change to read as follows:

6. The standard tempers of annealed material are as designated in Table III in the column entitled "Temper, Nominal Grain Size." Special or non-standard tempers are subject to negotiation between manufacturer and purchaser (Section 3 (*2*)).

Standard Specification for Copper Rod, Bar and Shapes (B 133 – 58):

Section 11.—Change the magnification requirements from "not less than 75 diameters" to read "from 75 to 200 diameters inclusive."

Standard Specification for Seamless Brass Tube (B 135 – 58):

Section 1.—Change the first sentence to read: "This specification covers seamless round and rectangular including square brass tube in straight lengths." In the second sentence change "Six alloys" to "Seven alloys." To the tabulation add alloy No. 7, with nominal composition of 90.0 per cent copper and 10.0 per cent zinc.

Section 2.—Renumber as Section 3.

Section 3.—Renumber as Section 2, and change the title from "Basis of Purchase" to "Ordering Information." In Item (*4*) insert the words "or distance between parallel surfaces" after the word "Diameter."

Table I.—Add alloy No. 7 with the following chemical requirements: copper, 89.0 to 91.0 per cent; lead, 0.05 per cent max; iron, 0.05 per cent max; and zinc, remainder. In footnote *a* add the words "or distance between outside parallel surfaces" after the word "diameter."

Section 5.—Add the following sentences: "Rectangular including square tubes shall normally be supplied only in drawn (general purpose) temper. When there is a need for light-drawn or hard-drawn tempers these may be supplied as agreed upon between the manufacturer and the purchaser."

Table II.—Change the third column heading to read: "Outside Diameter or Major Distance Between Outside Parallel Surfaces, in." Change the first sentence of footnote *a* to read: "Rockwell hardness values shall apply only to tubes having a wall thickness of 0.012 in. or over and to round tubes having an inside diameter of $\frac{5}{16}$ in. or over and to rectangular including square tubes having an inside major distance between parallel surfaces of $\frac{3}{16}$ in. or over." Add a new footnote *b* to read: "[b] Light-drawn and hard-drawn tempers are normally available in round tube only," referencing it to the words "Light-drawn" and "Hard-drawn" in the second column.

Add alloy No. 7 with mechanical property requirements as shown in the accompanying Table I.

Table III.—Delete the parentheses around the word "Annealed" in the second column heading. Change the first sentence of footnote *a* to read: "Rockwell hardness values shall apply only to

TABLE I.—ADDITIONS TO TABLE II, SPECIFICATION B 135.

Alloy No.	Temper	Outside Diameter, in.	Wall Thickness, in.	Tensile Strength, psi	Rockwell Hardness, 30T
7	Drawn (general purpose)	all	all	40 000 min	38 min
	Hard-drawn	up to 1 incl	0.020 to 0.120 incl	52 000 min	55 min
		over 1 to 2 incl	0.035 to 0.180 incl		
		over 2 to 4 incl	0.060 to 0.250 incl		

TABLE II.—ADDITIONS TO TABLE III, SPECIFICATION B 135.

Alloy No.	Annealed Temper	Wall Thickness, in.	Rockwell Hardness		Average Grain Size, mm	
			Scale	Max	Min	Max
7	Soft anneal	up to 0.045 incl	30T	30	0.025	0.060
		over 0.045	F	70	0.025	0.060
	Light anneal	up to 0.045 incl	30T	37	[b]	0.035
		over 0.045	F	78	[b]	0.035

tubes having a wall thickness of 0.015 in. or over and to round tubes having an inside diameter of $\frac{5}{16}$ in. or over and to rectangular including square tubes having an inside major distance between parallel surfaces of $\frac{3}{16}$ in. or over."

Add alloy No. 7 with mechanical property requirements as shown in the accompanying Table II.

Section 7.—Change the second sentence to read: "Tension tests are required for tubes with a wall thickness under 0.020 in. and for round tubes having an inside diameter under $\frac{5}{16}$ in. and for rectangular including square tubes having a major distance between inside parallel surfaces under $\frac{3}{16}$ in."

Section 9.—Change the title to read: "Expansion Test for Round Tube."

Section 11.—Change Paragraphs (*b*), (*c*), and (*g*) and add a new Paragraph (*h*) to read as follows:

(*b*) *Wall Thickness Tolerances.*—See Section 8(*d*), Tables V and XI.

(*c*) *Tolerances for Diameter or Distance Between Parallel Surfaces.*—See Section 8(*e*), Tables VI and XII.

(*g*) *Straightness Tolerances.*—For round tubes see Section 8(*j*)(*1*), Table X. For rectangular including square tubes see Section 8(*j*)(*2*).

(*h*) *Corner Radius for Rectangular Including Square Tubes.*—See Section 8(*k*), Table XIII.

Standard Specification for Copper Sheet, Strip, Plate, and Rolled Bar (B 152 – 58):

Section 5.—Change to read as follows:

5. The standard tempers of cold-rolled sheet, strip, plate, and rolled bar copper of all types are designated as follows:

(Retain present tempers and footnote *a*)

Special or non-standard tempers are subject to negotiation between manufacturer and purchaser (Section 3 (*2*)).

Section 6.—Change to read as follows, retaining the present Note 2:

6. The standard tempers of cold-rolled annealed copper sheet and strip are designated as follows:

Soft anneal, and
Deep-drawing anneal.

Special or non-standard tempers are subject to negotiation between manufacturer and purchaser.

Section 12.—Change the magnification requirements from the present "not less than 75 diameters" to read "from 75 to 200 diameters, inclusive."

Standard Specification for Copper Bus Bar, Rod, and Shapes (B 187 – 58):

Section 7.—Change the magnification requirements from the present "not less than 75×" to read "from 75 to 200 diameters, inclusive."

Standard Specification for Seamless Copper Bus Pipe and Tube (B 188 – 59):

Section 2(c).—Change the title to read: "Average Diameter, for Round Tube Only."

Section 7.—Change the magnification requirements from the present "not less than 75 diameters" to read "from 75 to 200 diameters, inclusive."

Section 8.—Change the title to read: "Bend Tests." Change the third sentence to read: "Other tubular sections shall be bent around a mandrel having a radius equal to six times the section dimension radial to the bend."

Section 9.—Change the title to read: "Sampling and Number of Tests." Change the opening word of Paragraph (*a*) from "Specimens" to "Sample pieces." In the second column heading of the schedule in Paragraph (*a*) change "Specimens" to "Sample Pieces." Add the words "but not to exceed 10 sample pieces" to the last value in column 2 of the schedule in Paragraph (*a*), and delete footnote *a* and the reference to it.

In Paragraph(*b*) add the words "taken from each sample piece" to follow the word "Specimens" in the first line. Add at the end a new sentence to read: "The value for the Rockwell hardness number of each specimen shall be established by taking the arithmetical average of at least three readings."

In Paragraph (*c*), in the first line, add the words "taken from each sample piece" to follow the words "One specimen."

In Paragraph (*d*), in line 3 replace the word "specimen" with "sample piece."

TABLE III.—WALL THICKNESS TOLERANCES FOR COPPER RECTANGULAR AND SQUARE TUBE.

(New Table VII, Specification B 188)

NOTE.—Maximum deviation at any point. The following tolerances are plus and minus; if tolerances all plus or all minus are desired, double the values given.

Wall thickness, in.	Distance Between Outside Parallel Surfaces, in.[a]						
	1/32 to 1/8, incl	Over 1/8 to 5/8, incl	Over 5/8 to 1, incl	Over 1 to 2, incl	Over 2 to 4, incl	Over 4 to 7, incl	Over 7 to 10, incl
Under 0.018	0.002	0.002	0.0025	0.003	......	......	......
0.018 incl to 0.025	0.003	0.0025	0.003	0.0035	......	......	......
0.025 incl to 0.035	0.0035	0.0035	0.0035	0.004	0.006	......	......
0.035 incl to 0.058	0.004	0.004	0.0045	0.005	0.007	0.009	......
0.058 incl to 0.083	......	0.005	0.006	0.007	0.008	0.010	0.012
0.083 incl to 0.120	......	0.007	0.008	0.009	0.010	0.012	0.014
0.120 incl to 0.165	......	0.009	0.010	0.011	0.012	0.014	0.016
0.165 incl to 0.220	......	0.011	0.012	0.013	0.015	0.017	0.019
0.220 incl to 0.284	......	......	0.015	0.016	0.018	0.020	0.022

[a] In the case of rectangular tube the major dimension determines the thickness tolerance applicable to all walls.

Section 12.—In Paragraph (*b*) change the last part of the sentence, beginning with the words "a retest shall . . ." to read: "a retest on an additional specimen either from the same sample piece or from a new sample piece shall be allowed."

In Paragraph (*c*) change the last half of the sentence to read: "a retest shall be permitted on a new sample piece or on the remaining portion of the first sample piece."

Change Paragraph (*d*) to read as follows:

(*d*) If the results of the test on one of the specimens fail to meet the specified requirements, two additional specimens shall be taken from different sample pieces and tested. The results of the tests on both of these specimens shall meet the specified requirements. Failure of more than one specimen to meet the specified requirements for a particular property shall be cause for rejection of the entire lot.

Section 13.—Change Paragraph (*f*) to read as follows:

(*f*) *Copper Tube* (*Other than Pipe, Water Tube and Threadless Pipe Sizes:*

(*1*) *Round Tube.*—Wall thickness tolerances shall be in accordance with Table V. Diameter tolerances shall be in accordance with Table VI.

(*2*) *Rectangular Including Square Tube.*—Wall thickness tolerances shall be in accordance with Table VII. The tolerances on distance between parallel surfaces shall be in accordance with Table VIII.

Change Paragraph (*l*) to read as follows:

(*l*) *Squareness of Cut.*—The angle of cut of the end of the pipe or tube furnished in straight lengths may depart from square by not more than the amounts shown in Table XII.

Delete Paragraph (*n*), relettering subsequent paragraphs accordingly.

Add a new Paragraph (*p*) to read as follows:

"(*p*) *Corner Radius.—Rectangular and Square Tubes.*—The permissible radii for commercially square corners shall be in accordance with Table XIII."

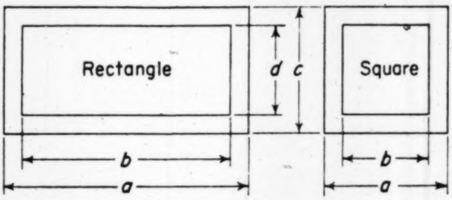
Table VII.—In the column headings replace the words "Outside Diameter" with the words "Major Outside Dimensions." Renumber as Table IX.

TABLE IV.—TOLERANCES OF DISTANCE BETWEEN PARALLEL SURFACES FOR COPPER RECTANGULAR AND SQUARE TUBE.

(New Table VIII, Specification B 188)

NOTE.—The following tolerances are plus and minus; if tolerances all plus or all minus are desired, double the values given.

Dimension *a* or *b* (see sketches), in.	Tolerances, in.
Up to ⅛, incl	0.003
Over ⅛ to ⅝, incl	0.004
Over ⅝ to 1, incl	0.005
Over 1 to 2, incl	0.006
Over 2 to 3, incl	0.007
Over 3 to 4, incl	0.008
Over 4 to 5, incl	0.009
Over 5 to 6, incl	0.010
Over 6 to 8, incl	0.011
Over 8 to 10 incl	0.012



Nominal dimension *a* determines tolerance applicable to both *a* and *c*:
Nominal dimension *b* determines tolerance applicable to both *b* and *d*.

Table VIII.—Change the column heading of the first column from "Outside Diameter, in." to read "Outside Diameter or Major Distance Between Outside Parallel Surfaces, in." Renumber as Table X.

New Tables.—Add new Tables VII and VIII for Section 13(*f*) (*2*) to read as shown in the accompanying Tables III and IV, renumbering present tables accordingly. Add new Tables XII and XIII for Section 13(*l*) and Section 13(*p*) to read as shown in the accompanying Tables V and VI.

Standard Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 - 59):

Table XV.—Revise as shown in the accompanying Table VII.

Standard Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 - 59):

Section 2(*c*).—Change the title to read: "Average Diameter, for Round Tubes Only."

Section 4(*c*).—Change to read as follows:

(*c*) *Physical and Mechanical Tests.*—For the physical and mechanical tests, unless otherwise provided in the product specification, a specimen shall be taken from each of the pieces selected in accordance with Paragraph (*a*). The required test shall be made on each of the specimens so selected. The value for the Rockwell hardness number of each specimen shall be established by taking the arithmetical average of at least three readings.

Section 5(*e*).—Change to read as follows:

(*e*) The surface of the test specimen for microscopic examination shall approximate a radial longitudinal section of round tube and a longitudinal section of rectangular and square tube perpendicular to, and bisecting, the major dimensional surface.

Section 7.—Change Paragraphs (*b*), (*c*), and (*d*) to read as shown above for Section 12(*b*), (*c*), and (*d*) of Specification B 188.

Section 8.—Add Item (*4*) to Paragraph (*a*) as shown in the tentative revision issued June, 1959, and appearing in the 1959 B-5 compilation, except start Item (*4*) as follows: "When round tube is ordered...."

Change Paragraph (*d*), Item (*3*) by the addition of the words "for round tube" after "designations B 68, B 75, and B 135," and add the following new sentence: "Wall thickness tolerances for rectangular including square tube applicable to B 75 and B 135 shall be in accordance with Table XI."

TABLE V. — TOLERANCES FOR SQUARENESS OF CUT FOR COPPER RECTANGULAR AND SQUARE TUBE.

(New Table XII, Specification B 188)

Outside Diameter or Major Distance Between Outside Parallel Surfaces, in.	Maximum Departure from Square, in.	
	For Pipe and Round Tube	For Square and Rectangular Tube
Up to 5/8 incl..	0.010	0.016
Over 5/8.......	0.016 per in. of diameter	0.025 per in. of distance between parallel surfaces

TABLE VI.—PERMISSIBLE RADII FOR COMMERCIALLY SQUARE CORNERS FOR COPPER AND COPPER-ALLOY RECTANGULAR AND SQUARE TUBE.

(New Table XIII, Specification B 188)

Wall Thickness, in.	Maximum radii, in.	
	Outside Corners	Inside Corners
Up to 0.058, incl..	3/64	1/32
Over 0.058 to 0.120, incl......	1/16	1/32
Over 0.120 to 0.250, incl......	3/32	1/32
Over 0.250.......	none established	none established

Change the heading of Paragraph (*e*) to read: "Diameter or Distance Between Parallel Surfaces, Tolerances." Change Paragraph (*e*), Item (*2*) by the addition of the words "for round tube" after "designations B 68, B 75, and B 135," and add the following new sentence: "Tolerances on distance between parallel surfaces for rectangular including square tube applicable to ASTM B 75 and B 135 shall be in accordance with Table XII."

Add a new sentence to Item (*3*) of Paragraph (*h*) as shown in the tentative revision issued June 1959 and appearing in the 1959 B-5 compilation.

TABLE VII.—STRAIGHTNESS TOLERANCES FOR ROD, BAR, AND SHAPES.

(Revision of Table XV, Specification B 249)

(Applicable to ASTM Designations B 16, B 21, B 98, B 133, B 138, B 139, B 140, B 150, B 151, and B 196)

	Length, ft	Maximum Curvature (Depth of Arc), in.
Drawn rod....	Up to 2	1/32
	incl 2 to 5	1/32 in any 2-ft portion[a]
	incl 5 to 10	1/8 in any 5-ft portion[a]
	10 and over	1/2 in any 10-ft portion[a]
Bar and shape (rolled or drawn)......	6 and over	1/2 in any 6-ft portion[a, b]

[a] Of total length.

[b] Applicable to any longitudinal surface or edge.

Change Paragraph (*i*) to read as follows:

(*i*) *Squareness of Cut* (*Applicable to ASTM Designations B 42, B 43, B 68, B 75, B 88, and B 135*).—The angle of cut of the end of the pipe or tube furnished in straight lengths may depart from square by not more than the following amounts:

Outside Diameter or Major Distance Between Outside Parallel Surfaces, in.	Maximum Departure from Square, in.	
	For Pipe and Round Tube	For Square and Rectangular Tube
Up to 5/8 incl..	0.010	0.016
Over 5/8.......	0.016 per inch of diameter	0.025 per inch of distance between parallel surfaces

To Paragraph (*j*) add a new Item (*2*) to read as follows:

(*2*) *Rectangular and Square Tubes.*—For rectangular and square tubes of any drawn temper, the straightness tolerance applicable to ASTM designations B 75 and B 135 shall be ½ in. maximum curvature (depth of arc) in any 6-ft portion of the total length. (Not applicable to extruded tube, redraw tube, or any annealed tube.)

Add a new Paragraph (*k*) to read as follows:

(*k*) *Corner Radius:*

(*1*) *Rectangular and Square Tubes.*—The permissible radii for commercially square corners applicable to ASTM Designations B 75 and B 135 shall be in accordance with Table XIII.

Table V.—Add a footnote to Table V as shown in the tentative revision issued June 1959 and appearing in the 1959 B-5 compilation except start the footnote as follows: "When round tube is ordered . . . ."

Table VII.—In the column headings change the words "Outside Diameter" to read: "Major Outside Dimensions."

Table IX.—Change the first column heading from "Outside Diameter, in." to read: "Major Outside Dimensions, in."

New Tables.—Add new Tables XI, XII, and XIII. Table XI is to be entitled "Wall Thickness Tolerances for Copper and Copper-Alloy Rectangular and Square Tube (Applicable to ASTM Designations B 75 and B 135)," and otherwise to be as shown above for Table VII, Specifications B 188. Table XII is to be entitled "Tolerances on Distance Between Parallel Surfaces for Copper and Copper-Alloy Rectangular and Square Tube (Applicable to ASTM Designations B 75 and B 135)," and otherwise to be as shown above for Table VIII of Specifications B 188. Table XIII is to read as shown in the accompanying Table VIII.

TABLE VIII.—PERMISSIBLE RADII FOR COMMERCIALLY SHARP CORNERS FOR COPPER AND COPPER-ALLOY RECTANGULAR AND SQUARE TUBE.
(Applicable to ASTM Designations B 75 and B 135)
(New Table XIII for Specification B 251)

Wall Thickness, in.	Maximum Radii, in.	
	Outside Corners	Inside Corners
Up to 0.058, incl. .	3/64	1/32
Over 0.058 to 0.120, incl.	1/16	1/32
Over 0.120 to 0.250, incl.	3/32	1/32
Over 0.250.	none established	none established

Standard Specification for Seamless Copper Tube for Refrigeration Field Service (B 280 – 59):

Section 9.—Add the following sentence: "When DHP copper is supplied, microscopic examination for cuprous oxide is not required."

Section 10.—Change the magnification requirements from the present "not less than 75 diameters" to read: "from 75 to 200 diameters, inclusive."

Section 17(*b*).—Delete the last part of the first sentence, beginning with the words "except for the nominal . . . ."

Table II.—Delete Footnote *a* and the references thereto. Change the figures opposite the ¾ and ⅞ in. sizes in the fourth and fifth columns to read as follows:

¾.	8.72	0.0305
⅞.	10.28	0.0360

Standard Specification for Threadless Copper Pipe (B 302 – 59):

Section 13(*e*).—Change the end of the first sentence to read: ". . . except for the 12-in. size which shall be 15 ft."

Table I.—In column 4, change "115.4" to read "115."

Section 9(*c*).—Change the last word "table" to read "tube."

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standard specifications:

Standard Specification for Seamless Copper Pipe, Standard Sizes (B 42 – 58),

Standard Specification for Seamless Red Brass Pipe, Standard Sizes (B 43 – 58),

Standard Specification for Seamless Copper Tube (B 75 – 59),

Standard Specification for Seamless Copper Water Tube (B 88 – 58),

Standard Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 58),

Standard Specification for Threadless Copper Pipe (B 302 – 59),

Standard Specification for Copper Drainage Tube (DWV) (B 306 – 59), and

Standard Specification for Copper-Silicon Alloy Seamless Pipe and Tube (B 315 – 59):

Section on Hydrostatic Test.—Delete the word "unit" from the definition for "S."

Standard Specification for Phosphor Bronze Rod, Bar, and Shapes (B 139 – 58):

Table I.—For Alloy B1, change the minimum lead content from "0.08" to read "0.8."

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS*

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: in Atlantic City, N. J., on June 23, 1959, and in Chicago, Ill., on Feb. 3, 1960.

The committee consists of 86 members, of whom 41 are classified as producers, 20 as consumers, and 25 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. Babington.
Vice-Chairman, A. E. Weiss.
Secretary, G. L. Werley.
Advisory Committee Members-at-Large: D. L. Colwell, R. F. Cramer, E. N. Jacobi, J. W. Meier, A. J. Prickett, and D. L. La Velle.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Aluminum-Alloy Die Castings (**B 85 – 58 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Table I.—Change the maximum per cent of zinc from the present "1.0" to read "3.0" in both alloys SC84A and SC84B.

Table III.—Add the following values for alloy G8A:

Tensile strength, psi	45 000
Yield strength (0.2 per cent offset), psi	28 000
Elongation in 2 in., per cent	5
Shear strength, psi	29 000
Fatigue strength (R. R. Moore specimen), 500,000,000 cycles, psi	20 000

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specification for:

Zinc-Base Alloy Die Castings (**B 86 – 57 T**),[1] and
Zinc-Base Alloys in Ingot Form for Die Castings (**B 240 – 57 T**).[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. V. Blackmun, chairman) prepared the revisions in Specification B 85 covered earlier in this report, and recommended that the revised specification be adopted as standard.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) recommended that Specifications B 86 and B 240 be retained as tentative without revision.

A report covering a study of the mechanical properties of zinc alloys cast in evacuated dies in comparison with the properties of regularly produced die-cast test bars is appended.[3]

Subcommittee V on Exposure and Corrosion Tests (D. H. Kleppinger, chairman).—The 20-year aged zinc and magnesium alloy test bars have been successfully returned from the Coco Solo exposure in the Canal Zone due mainly to the efforts of L. B. Sartain of the Canal Zone.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 2.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] See p. 162.

The subcommittee also prepared the report covering the exposure tests on SC84A aluminum-alloy die-cast test bars with varying zinc contents which is appended.[4]

This report has been submitted to letter ballot of the committee, which consists of 86 members; 81 members returned their ballots, of whom 74 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. Babington,
Chairman.

George L. Werley.
Secretary.

---

[4] See p. 165.

APPENDIX I

MECHANICAL PROPERTIES OF ZINC ALLOY TEST BARS PRODUCED IN EVACUATED DIES

REPORT OF TASK GROUP OF SUBCOMMITTEE II ON ZINC-BASE DIE-CASTING ALLOYS

This task group was appointed in June, 1957, to investigate the mechanical properties of zinc die-castings made under vacuum conditions on commercial equipment.

Five sources for test bars were planned, but only three sources could be located. The vacuum system used was the Reed-Prentice or Nelmore system which completely encloses the die within a hood connected to a large accumulator. As the hood closes just prior to the closing of the die, a valve between accumulator and hood is opened and equilibrium vacuum is established between the accumulator and the hood. The vacuum within the die is the same as the vacuum in the hood.

Eighty tension test bars and 80 impact bars were made by each source, alternately producing 20 bars by vacuum and non-vacuum from the same heat of metal, with all other casting variables held constant.

Table I compares the production data for the three Suppliers A, B, and C. Suppliers A and B used a die supplied by The New Jersey Zinc Co. Supplier C used a larger die suitable for their large machine. As 6 months delay was encountered between the first and the third runs of test bars, all bars were aged for 6 months before being submitted to the testing laboratories.

All bars were permanently identified before cleaning and were radiographed to compare quality and to inspect for major defects. All tension test bars were radiographically sound in the reduced section. The impact bars from supplier C showed slight porosity, the porosity being slightly greater in the non-vacuum than in the vacuum bars. From a radiographic standpoint the test bars were all of good quality and none was discarded.

Table II summarizes the mechanical properties obtained by laboratories X, Y, and Z. Each value is the average of 10 bars submitted to each laboratory. The grand average of all producers is as follows:

	Vacuum	Non-Vacuum
Tensile strength, psi....	41 000	40 500
Elongation in 2 in., per cent...............	17	15
Impact strength, ft-lb...	41	39

After reviewing the data, it is the conclusion of the Task Group that there are minor but consistent differences in mechanical properties in favor of the vacuum die-casting system at the level of vacuum represented by the process used to produce the test bars.

In this respect the results do not confirm the claims which have been made indicating substantially superior mechanical properties for zinc die-castings made under vacuum conditions in the order of 18 to 22 in. of vacuum.

The report does not in any way reflect

on the value of the vacuum system as a means of improving the performance of a die or for improving the reliability of a system. The efforts of the committee were focused on determining whether improved mechanical properties could be obtained by die-casting in a die operated in a vacuum available on commercial machines. The results suggest a possibility of further improvement with increased vacuum level.

TABLE I.—DATA ON VACUUM CAST TEST BARS.

	Producer		
	A	B	C
Size machine	400 ton	400 ton	500 ton
Vacuum system	R-P	R-P	R-P
Accumulator vacuum, in.	27	21	25
Hood vacuum at shot, in.	21	17	20.5
Casting cycle, sec	22	10.6	13
Pressure, psi:			
Line	1050	1180	1200
Metal	2370	5980	2550
Cylinder to plunger ratio	4½ to 3	...	4 to 2¾
Metal temperature, deg Fahr	790	780	780
Alloy	AG40A	AG40A	AG40A
Date of Run	5-12-58	8-8-58	11-20-58
Witnessed by	Hodgson Muenger	None None	Hodgson Muenger

	Quantometric Analysis, Element, per cent					
	Lead	Magnesium	Aluminum	Copper	Tin	Cadmium
Producer A	0.0023	0.03	3.60	0.14	0.0010	0.0010
Producer B	0.0020	0.032	4.15	0.06	0.0008	0.0008
Producer C	0.0024	0.036	3.90	0.10	0.0007	0.0009

It is recommended by the committee that further studies of castings or test bars produced at higher levels of vacuum be made as soon as systems are available commercially to reach higher levels of vacuum.

The possible beneficial effects of casting under vacuum on the quality of die-cast aluminum should also be suggested for investigation in the future when more commercial experience is available.

Respectfully submitted by the Task Group,

G. F. Hodgson, *Chairman*
W. Babington
C. R. Muenger
M. R. Tenenbaum
G. L. Werley

TABLE II.—SUMMARY SHEET.

	Producer A			Producer B			Producer C		
	Laboratory X	Laboratory Y	Laboratory Z	Laboratory X	Laboratory Y	Laboratory Z	Laboratory X	Laboratory Y	Laboratory Z
VACUUM									
Tensile strength, psi	40 300	38 600	40 800	41 000	40 400	43 550	42 400	42 020	40 420
Yield strength, psi	...	30 000	...	...	32 000	...	...	31 380	...
Impact, ft-lb:									
Gate	44	35.8	37.6	50	39.2	38.8	37	37.0	36.6
Vent	48	39	42.0	48	41.4	40.4	40	36.7	40.9
Brinell hardness	74	...	...	74	...	...	75	...	...
Elongation in 2 in., per cent	20	19.9	18.8	18	16.5	20.7	14	13.6	14.6
Average:									
Tensile strength, psi		39 900			41 600			41 600	
Yield strength, psi		30 000			32 000			31 380	
Elongation in 2 in., per cent		20			18			14	
Impact, ft-lb:									
Gate		39			43			37	
Vent		43			43			39	
NON-VACUUM									
Tensile strength, psi	40 200	38 500	40 300	41 000	40 200	40 940	42 000	41 290	40 320
Yield strength, psi	...	30 500	...	...	32 000	...	...	31 390	...
Impact, ft-lb:									
Gate	38	38.8	36.3	49	39.4	40.0	33	36.9	29.8
Vent	45	40.2	40.4	48	41.2	39.9	32	33.9	33.3
Brinell hardness	74	...	...	74	...	...	74	...	...
Elongation in 2 in., per cent	15	15.5	12.9	18	17.1	16.3	12	12.8	13.2
Average:									
Tensile strength, psi		39 600			40 700			41 200	
Yield strength, psi		30 500			32 000			31 390	
Elongation in 2 in., per cent		14			17			13	
Impact, ft-lb:									
Gate		38			43			33	
Vent		42			43			33	

APPENDIX II

EXPOSURE TESTS OF SC84A ALUMINUM DIE-CAST TEST BARS WITH VARYING ZINC CONTENTS

Report of Subcommittee V on Exposure and Corrosion Tests

TABLE I.—MECHANICAL PROPERTIES OF CONTROLLED ZINC BARS AS-CAST AND AFTER 1- AND 3-YR EXPOSURE.

	Tensile Strength, psi			Yield Strength, psi			Elongation in 2 in., per cent		
As-cast:									
Alloy 1 (0.25 Zn)	41 700			20 400			3.4		
Alloy 2 (0.50 Zn)	41 500			20 100			3.5		
Alloy 3 (1.0 Zn)	41 900			20 100			3.7		
Alloy 4 (1.5 Zn)	42 100			20 200			3.8		
Alloy 5 (2.0 Zn)	42 600			20 200			4.0		
Alloy 6 (3.0 Zn) (Made only by one producer)	41 800			20 200			3.9		
	New York City	Kure Beach 80-ft	Kure Beach 800-ft	New York City	Kure Beach 80-ft	Kure Beach 800-ft	New York City	Kure Beach 80-ft	Kure Beach 800-ft
After 1 yr:									
Alloy 1	42 500	37 400	38 500	23 600	23 500	23 200	2.9	1.9	2.0
Alloy 2	40 800	36 200	38 000	23 400	22 900	23 100	2.8	1.9	1.9
Alloy 3	42 400	36 100	36 600	23 600	22 900	23 400	3.1	1.8	2.1
Alloy 4	42 000	36 200	37 400	23 400	23 100	22 900	3.0	1.8	2.0
Alloy 5	41 700	36 900	36 800	23 200	23 300	23 000	3.0	2.0	1.8
Alloy 6	42 500	35 300	36 500	23 900	23 300	23 200	3.0	1.9	1.9
After 3 yr:									
Alloy 1	41 600	36 900	37 700	27 500	24 800	23 600	2.3	1.8	1.9
Alloy 2	39 900	39 000	37 300	23 500	24 700	23 000	2.3	2.1	2.0
Alloy 3	42 400	[a]	37 100	25 100	[a]	23 100	3.1	[a]	2.0
Alloy 4	40 200	35 400	36 200	23 400	22 900	23 100	2.5	2.0	1.8
Alloy 5	41 500	[a]	36 600	24 500	[a]	23 200	2.6	[a]	1.9
Alloy 6	...	[a]	35 900	[a]	[a]	23 200	[a]	[a]	1.7

[a] Bars lost in Hurricane Hazel.

At the June 1949 meeting of Committee B-6, a proposal was made to increase the zinc content of the SC84 alloy from 0.6 to 1.0 per cent. Before approving this change, the committee decided to make tests on the SC84A alloy with varying zinc contents. The five zinc contents chosen for study were 0.25, 0.50, 1.0, 1.5, and 2.0 per cent. The description of the production and testing of the test bars as well as the results of these tests are given in Appendix I of the 1953 report of Committee B-6.[1]

At the 1953 meeting of Committee

---

[1] "The Effect of Zinc Content on the Mechanical Properties of the SC84A Aluminum Die-Casting Alloy," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 212 (1953).

TABLE II.—PORT AUTHORITY BUILDING, 6-YR EXPOSURE.

Number[a]	Test Laboratories									Average		
	X			Y			Z					
	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent
A_1.......	36 900	23 200	2.0	39 700	22 500	2.4	36 900	22 900	2.0	39 400	22 700	2.2
				43 200	21 600	2.8	40 100	23 500	2.0			
B_1.......	43 600	24 200	2.5	44 300	23 200	3.0	41 500	23 100	2.0	42 600	23 500	2.5
	44 400	24 400	2.5				39 300	22 700	...			
C_1.......	43 100	26 300	2.5	43 000	25 700	2.8	40 500	25 500	2.5	42 300	25 700	2.5
	43 000	25 500	2.5				42 000	25 300	2.0			
							Grand average.....			41 400	24 000	2.4
A_2.......	41 800	24 000	2.5	38 100	22 900	2.6	39 300	22 800	2.0	40 100	23 000	2.4
	42 000	23 600	2.5				39 500	21 900	...			
B_2.......	41 100	23 500	2.5	41 300	22 800	2.8	41 600	22 900	2.5	41 600	23 200	2.5
	43 000	23 600	2.5	40 900	23 000	2.4						
C_2.......	42 600	25 900	2.5	43 800	25 300	3.0	sample missing			42 000	25 300	2.7
	42 200	25 400	2.5	39 400	24 400	...						
							Grand average.....			41 200	23 800	2.5
B_3.......	44 700	24 100	3.0	42 500	23 200	2.8	39 800	23 100	2.0	41 400	23 200	2.4
				41 600	22 400	2.4	38 400	23 100	2.0			
C_3.......	41 200	24 200	2.5	43 900	25 100	3.5	40 300	24 900	...	41 300	24 300	3.0
				42 600	24 200	3.0	38 600	22 400	...			
							Grand average.....			41 400	23 800	2.7
A_4.......	41 200	23 400	2.5	42 000	22 800	2.8	40 700	27 800	...	42 100	24 200	2.6
	45 000	24 500	2.5	41 800	22 400	2.5						
B_4.......	40 900	23 400	2.5	42 700	23 500	3.0	40 600	22 500	2.0	40 700	23 200	2.3
	41 600	23 800	2.0				37 600	22 600	2.0			
C_4.......	41 600	24 500	2.5	41 800	24 900	...	42 400	24 300	1.0	41 200	24 600	2.0
	41 200	24 500	2.5				39 200	24 600	2.0			
							Grand average.....			41 300	24 000	2.3
A_5.......	37 100	22 000	2.2	42 100	23 200	2.6	46 400	23 600	2.5	41 400	23 300	2.4
				40 700	24 200	2.4	40 600	23 600	...			
B_5.......	43 400	24 000	3.0	43 300	23 200	2.7	40 700	22 400	...	42 300	23 100	2.8
	42 300	23 200	2.5	41 900	22 600	2.8						
C_5.......	42 700	22 700	3.0	42 400	25 100	2.8	38 200	23 700	...	41 300	24 300	2.8
	42 600	24 700	2.5	40 800	25 100	...						
							Grand average.....			41 700	23 600	2.7
B_6.......	42 200	24 600	2.0	43 700	23 900	2.4	41 200	23 600	...	41 800	23 900	2.2
				41 900	23 800	2.5	40 200	23 800	2.0			
							Grand average.....			41 800	23 900	2.2

---

[a] Bars identified as follows:

A, B, and C—Producer code, and subscript numbers indicate percentage of zinc:

1 = 0.25	4 = 1.50.
2 = 0.50	5 = 2.00.
3 = 1.00	6 = 3.00.

TABLE III.—KURE BEACH, 800-FT SITE, 6-YR EXPOSURE.

Number[a]	Test Laboratories									Average		
	X			Y			Z					
	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent
A_1	39 600	22 800	2.0	36 300	21 500	2.2	39 100	23 900	2.0	38 800	22 800	2.1
	41 100	23 500	2.0	37 800	22 400	2.2						
B_1	42 200	24 300	2.0	39 000	23 500	2.0	38 100	23 000	1.5	39 400	23 500	2.0
				40 100	23 300	2.3	37 400	23 600	2.0			
C_1	38 800	24 800	2.0	41 900	25 000	2.4	35 900	23 600	2.0	38 900	24 300	2.0
				39 600	24 300	2.1	38 500	23 600	1.5			
							Grand average.....			39 000	23 500	2.0
A_2	29 300	21 100	1.0	36 400	23 100	1.6	37 800	22 800	2.0	34 600	22 300	1.4
	32 900	22 200	1.0				36 700	22 200	1.5			
B_2	37 400	22 800	1.5	38 100	22 500	1.6	38 100	...	2.0	37 900	23 100	1.7
	37 700	24 100	1.5	38 400	22 900	1.9						
C_2	36 200	23 800	2.0	39 000	25 100	1.5	37 900	24 700	1.5	38 000	25 100	1.6
	36 600	25 500	1.0	40 100	26 400	2.0						
							Grand average.....			36 800	23 500	1.6
A_3	......	......	......	37 700	22 700	1.8	33 700	21 300	1.5	35 000	21 600	1.6
							33 500	20 900	1.5			
B_3	39 500	23 400	2.0	38 500	22 800	1.6	39 900	23 800	1.0	38 700	23 600	1.6
	36 900	23 400	1.5				38 500	24 800	2.0			
C_3	37 600	25 500	2.0	39 500	25 300	1.6	37 400	24 200	1.0	38 800	25 300	1.6
	40 000	28 100	1.5				39 500	23 500	2.0			
							Grand average.....			37 500	23 500	1.6
A_4	37 900	23 500	1.5	35 700	22 300	1.9	37 000	22 400	2.5	34 700	22 500	1.8
	25 900	21 600	1.0	36 900	22 500	2.0						
B_4	37 700	23 600	1.5	37 500	22 700	1.8	37 700	22 000	3.5	37 800	22 800	1.8
				38 900	23 200	2.0	37 100	22 200	1.0			
C_4	37 100	23 900	1.5	35 400	23 700	1.8	37 600	22 500	2.5	37 100	23 400	1.9
				38 200	24 700	2.0	37 000	22 300	1.5			
							Grand average.....			36 500	23 900	1.9
A_5	35 200	23 400	1.0	35 900	23 400	1.7	37 200	22 700	2.0	36 400	23 200	1.6
	35 800	23 400	1.5				37 700	23 100	2.0			
B_5	36 900	22 900	1.5	36 800	23 100	1.6	37 300	22 400	2.0	37 300	23 000	1.7
	37 800	23 600	1.5	37 500	22 800	1.8						
C_5	37 700	24 200	2.0	35 600	23 700	1.5	37 600	22 500	2.5	37 200	23 500	1.9
				38 300	23 700	1.8	37 000	23 300	1.5			
							Grand average.....			37 000	23 500	1.9
B_6	36 900	23 500	1.5	37 500	23 000	1.9	36 500	22 500	1.5	37 100	23 100	1.6
	36 100	23 100	1.5				38 300	23 300	1.5			
							Grand average.....			37 100	23 100	1.6

[a] Bars identified as follows:

A, B, and C—Producer code, and subscript numbers indicate percentage of zinc:

1 = 0.25. 4 = 1.50.
2 = 0.50. 5 = 2.00.
3 = 1.00. 6 = 3.00.

B-6 a series of exposure tests was initiated in which the excess bars from the zinc study were utilized. Bars were exposed at the Kure Beach 80-ft site for 1 and 3 yr; at the Kure Beach 800-ft site for 1, 3, and 6 yr; and on the New York Port Authority Roof for 1, 3, 6, and 12 yr. Test bars were exposed in April 1953. One-year test bars were removed from exposure in 1954 and 3-yr bars were removed in 1956. The results of the 1- and 3-year tests were published as an appendix to the report of Committee B-6 for 1957.[2] For convenience in evaluating the data in this report, the mechanical properties of test bars as-cast and after 1 and 3 yr of exposure are shown in Table I.

In April 1959, the 6-yr test bars were withdrawn from the New York Port Authority Roof and from the Kure Beach 800-ft site. The bars were tested at the Aluminum Co. of America, the Pitman-Dunn Laboratories of Frankford Arsenal, and at the Bell Telephone Laboratories. The individual test values as well as the averages for these bars are listed in Tables II and III.

In Table IV, the percentage differences between the as-cast mechanical properties and the properties after the exposure times of 1, 3, or 6 yr for the respective exposure sites are shown.

TABLE IV.—CHANGES IN MECHANICAL PROPERTIES AFTER VARIOUS EXPOSURE PERIODS.

Alloy Number	New York			Kure Beach, 80-ft			Kure Beach, 800-ft		
	1 yr	3 yr	6 yr	1 yr	3 yr	6 yr	1 yr	3 yr	6 yr
PER CENT CHANGE IN TENSILE STRENGTH									
1	+1.9	−0.2	−0.72	−10.3	−11.5	...	−7.7	−9.6	−6.5
2	−1.7	−3.9	−0.72	−12.8	−6.0	...	−8.2	−10.1	−11.3
3	+1.2	+1.2	−1.17	−13.8	[a]	...	−12.7	−10.0	−10.4
4	−0.2	−4.5	−1.90	−14.1	−15.9	...	−11.2	−14.0	−13.3
5	+1.2	−2.6	−0.95	−13.4	[a]	...	−13.6	−14.1	−13.1
6	+1.7	[a]	0	−15.5	[a]	...	−12.7	−14.1	−11.3
PER CENT CHANGE IN YIELD STRENGTH									
1	+15.7	+34.8	+17.6	+15.2	+21.6	...	+12.7	+15.7	+15.2
2	+16.4	+16.9	+18.4	+12.9	+22.8	...	+14.9	+14.4	+16.9
3	+17.4	+24.9	+18.4	+12.9	[a]	...	+16.4	+14.9	+16.9
4	+15.9	+15.8	+18.8	+14.4	+13.4	...	+13.4	+14.3	+18.3
5	+14.9	+21.3	+16.8	+15.4	[a]	...	+13.9	+14.8	+16.3
6	+18.3	[a]	+18.3	+15.4	[a]	...	+14.9	+14.8	+14.4
PER CENT CHANGE IN ELONGATION									
1	−15	−32	−29	−44	−47	...	−41	−44	−41
2	−20	−34	−29	−46	−40	...	−46	−43	−54
3	−16	−16	−27	−51	[a]	...	−43	−46	−59
4	−21	−34	−39	−53	−47	...	−47	−53	−50
5	−25	−35	−33	−50	[a]	...	−55	−52	−53
6	−20	[a]	−44	−51	[a]	...	−51	−59	−59

[a] Bars lost in Hurricane Hazel, 1954.

---

[2] "Exposure Tests of SC84A Aluminum Bars with Varying Zinc Content," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 174 (1957).

Analysis of the data leads to confirmation of the facts that (1) alloy SC84 undergoes precipitation hardening as evidenced by the increased yield strength in all aged alloys and by the loss of ductility; and (2) alloy SC84 with zinc content from 0.25 to 3.0 per cent without any corrosion protective treatment withstands the New York City environment without any appreciable loss of tensile properties. These data also show that the SC84 alloy with zinc content at 0.25 per cent shows the best resistance to atmospheric exposure, but that there is no significant difference in resistance to corrosion between alloys in which the zinc content ranges between 0.5 and 3.0 per cent.

The withdrawal and testing of the 12-yr bars from the New York Port Authority Building Roof in 1965 will complete this program.

The cooperation extended by the Precision Casting Co., Bell Telephone Laboratories, and Stewart Warner Co. in producing the tension bars and of the Aluminum Co. of America, Frankford Arsenal, and Bell Telephone Laboratories in performing tension tests is gratefully acknowledged by Subcommittee V.

Respectfully submitted on behalf of the subcommittee,

D. H. KLEPPINGER,
Chairman.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys, Cast and Wrought, held two meetings during the year: at Atlantic City, N. J., in June, 1959, and at Chicago, Ill., in Feb., 1960.

The committee consists of 90 members of whom 90 are voting members; 37 are classified as producers, 34 as consumers, and 19 as general interest members.

I. V. Williams resigned as chairman of Committee B-7 in order to devote more time to other ASTM activities, but he will continue to be a member of the committee. As chairman since 1946, he has given the committee excellent leadership which is appreciated by all members. R. A. Harris was elected to fill the unexpired term, and resigned as chairman of Subcommittee IX, being replaced by J. J. Martin. F. M. Barry, J. P. Brinn, and J. S. Laird resigned from the committee because of retirement, and C. F. Nixon and R. A. Quadt resigned because of increased responsibilities. Appointment of the following representatives to other ASTM committees was made: C. R. Mielke to Subcommittee II of Committee E-7 on Nondestructive Testing; and R. A. Harris to replace I. V. Williams on the Coordinating Committee on Non-Ferrous Metals and Alloys and on the Advisory Committee on Corrosion.

Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys has been transferred to Committee B-8 on Electrodeposited Metallic Coatings.

The Regulations Governing Committee B-7 were revised, effective Jan. 8, 1960, to clarify voting, types of members, and classifications of interest, and to add the normal procedure and an alternate procedure for recommendations affecting standards and tentatives.

The Advisory Committee continued to function as advisor to the American Standards Assn. in its representation of the United States as a participating member of Technical Committee 79 on Light Metals and Their Alloys of the International Organization for Standardization. The following ISO Recommendations have been accepted by the ISO Council for publication:

R114 on Composition of 99.8 Unalloyed Magnesium Ingots,
R115 on Classification and Composition of Remelt Ingots and Pigs of Unalloyed Aluminum,
R121 on Composition of Magnesium-Aluminum-Zinc Alloy Castings, and
R122 on Composition of Magnesium-Aluminum-Zinc Alloy Ingots for Casting Purposes.

The Advisory Committee recommended voting affirmatively on ISO Recommendations No. 325 on Composition of 99.95 Unalloyed Magnesium Ingots and No. 326 on Composition of Aluminum Alloy Castings, and negative on No. 327 on Composition of Wrought Products of Aluminum and Aluminum Alloys since it is in conflict with the composition limits in ASTM specifications.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. A. Harris.
Vice-Chairman, C. B. Gleason.
Vice-Chairman, D. L. Colwell.
Secretary, R. B. Smith.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends one new specification for publication as tentative, revisions in 16 tentative specifications, adoption of one tentative as standard with revision, and revision of 7 standards for immediate adoption. The standards and tentatives affected, together with the revisions recommended, are covered in detail in Appendix I to this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (D. L. LaVelle, chairman) revised the Standard Specification for Aluminum for Use in Iron and Steel Manufacture (B 37 – 59), the Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 59), and the Tentative Specification for Aluminum Alloy Hardeners Used in Making Zinc-Base Die Casting Alloys (B 327 – 58 T) to exempt the manufacturer from sampling and analyzing the finished product if he has made an analysis of the material during the course of manufacture. The subcommittee further revised Specification B 179 to add alloys SC51B and SG70B; and to increase the iron limit for alloys G8A, S12A-B, and SG100A-B, and the zinc limit for SC84A-B to be consistent with the limits for castings of these alloys. The revision of Specification B 327 is recommended for adoption as standard. These revisions are included in Appendix I to this report.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman) revised the Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 59 T) to add the additional temper -T7 for alloy SG70A, as shown in Appendix I to this report.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (H. D. Monsch, chairman) assisted the ASTM Coordinating Committee on Non-Ferrous Metals and Alloys in developing the new Tentative Specification for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings appended to this report.[3]

The subcommittee revised the specifications under its jurisdiction to delete alloy designations conforming to Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 59) and reference to it, leaving only alloy designations conforming to the American Standard Alloy Designation System for Wrought Aluminum (ASA No. : H35.1—1957) arranged in ascending numerical order. In addition, as shown in Appendix I to this report, the subcommittee revised the Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209 – 59 T) to add plate flatness tolerances and revise chemical requirements, tensile requirements, and tolerances to bring them into better agreement with the latest industry standards; Tentative

---

[1] See p. 174.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[3] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

Specification for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 59 T) to add the -T31 temper for alloy 6063; Tentative Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 59 T) to correct the temper -H38 for Alclad 5056 to make it read "-H393"; Tentative Specification for Aluminum-Alloy Extruded Bars, Rods and Shapes (B 221 – 59 T) to add tolerances for corner and filet radii; and Tentative Specification for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 – 59 T) to add new tempers and classes of tolerances.[4]

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (A. A. Moore, chairman) revised the Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 57) to add alloys 9990A and 9995A, which are used as reducing agents in the production of nuclear grade metals; and revised the Standard Specifications for: Magnesium-Base Alloy Sheet and Plate (B 90 – 59), Magnesium-Base Alloy Forgings (B 91 – 59), Magnesium-Base Alloy Bars, Rods and Shapes (B 107 – 59), and Extruded Magnesium-Base Alloy Tubes (B 217 – 59) to specify the minimum size of subsize tension test specimens. The revisions are included in Appendix I to this report.

Subcommittee V on Testing Light Metals (J. C. Millson, chairman) recommended revisions in Committee B-7 specifications to add minimum dimensions for subsize tension test specimens to the sections on "Test Specimens." The subcommittee is seeking assistance from Subcommittee 4 on Tension Testing of Committee E-1 on Methods of Testing in establishing equivalence of subsize flat specimens with the standard flat tension test specimen shown in Fig. 7 of Methods of Tension Testing of Metallic Materials (E 8 – 57 T), and in establishing standard sizes for subsize flat specimens.

Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought (C. B. Gleason, chairman) is revising the Recommended Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 – 56) to amplify it and to define some recently developed tempers, and the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 59) to exclude wrought aluminum and aluminum alloys.

Subcommittee VIII on Atmospheric Exposure Tests (L. H. Adam, chairman) has test specimens of 27 aluminum and 8 magnesium commercial alloys, cast and wrought, at five ASTM atmospheric exposure test sites: on top of the Port Authority Building, New York City, for industrial atmospheric conditions; at State College, Pa., for rural; at Kure Beach, N. C., for Atlantic Coast marine; at Point Reyes, Calif., for Pacific Coast marine; and at Freeport, Tex., for Gulf Coast marine atmospheric conditions. The data on exposure for six months, one year, and three years are complete and have been published in ASTM *Proceedings*.[5] Data obtained from cast aluminum and magnesium alloys exposed for five years are given in Appendix II to this report.[6] The wrought aluminum and magnesium alloy specimens exposed for five years are being tested.

---

[4] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

[5] Data for 6-month and 1-yr exposures: L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 284 (1955); L. H. Adam, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 270 (1954).

Data for 3-yr exposures: L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 194 (1958); L. H. Adam and Marie Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 289 (1959).

Subcommittee IX, Editorial (J. J. Martin, chairman) is preparing Recommendations on Form of ASTM Committee B-7 Specifications to supplement Proposed Recommendations on Form of ASTM Specifications, April, 1957, which have been approved as a guide, insofar as practical, for writing and revising B-7 specifications. Standardization of section titles, sequence, and wording, and form, titles, and headings of tables is being attempted.

Special Subcommittee on Light Metals for Nuclear Applications (W. G. Groth, chairman) cooperated with Subcommittee IV in developing requirements for high-purity magnesium in ingot form of a quality suitable for use as a reducing agent in the production of nuclear grade metals, as shown in the proposed revision of Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 57) included in Appendix I to this report.

This report has been submitted to letter ballot of the committee, which consists of 90 members; 70 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. A. HARRIS,
Chairman.

R. B. SMITH,
Secretary.

---

[6] L. H. Adam and M. Dougherty, "Atmospheric Exposures of Aluminum and Magnesium Sand and Permanent Mold Castings," see p. 186.

APPENDIX I

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards and tentatives covering light metals and alloys which are referred to earlier in this report. The standards and tentatives appear in their present form in the 1958 Book of ASTM Standards, Part 2, or in the 1959 Supplement to Book of ASTM Standards, Part 2.

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Factory Made Wrought Aluminum and Aluminum-Alloy Welding Fittings as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following 16 tentatives and their continuation as tentative:

Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 59 T):

Table II.—For alloy SG70A add the temper T7 (Solution treated and overaged) with the following tensile requirements:

Yield strength (0.2 per cent offset), min, psi	...
Tensile strength, min, psi	31 000
Elongation in 2 in., min, per cent	[c]

Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209 – 59 T):

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1100, 1060, 5050, 5005, 5454, 5086, 5083, 5456, 5052, 5652, 5154, 5254, 3003, Alclad 3003, 3004, and Alclad 3004, yield strength is not important."

Table I.—Delete both the ASTM and AA subheadings under the column "Alloy." Delete the ASTM alloy designations in the table and footnote *a*, renumbering subsequent footnotes accordingly and revising present footnote *b* to read: "[a] These alloy designations were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No.: H35.1—1957)." Change the maximum limit for zinc for alloys 5052 and 5652 from "0.20" to "0.10."

Table II.—Change the ASTM designation in the note at the top of the table to read "E 29 – 50." Delete the ASTM alloy designation and the designation "AA" from all subheadings, revising subheadings to read: "Alloy 1100," "Alloy 1060," and so on.

In the column headed "Thickness, in.," revise the figures "0.500" and "0.501" to read "0.499" and "0.500," respectively, in all cases where they appear in the data for alloys 1100, 1060, 2024, 5005, 5086, 5052, 5652, 5154, 5254, 6061, 3003, and 7075.

---

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

For alloy 5086-O in the column headed "Thickness, in," revise the figure "0.250" to read "0.188."

For alloys 5083 and 5456, revise the tensile requirements to read as shown in the accompanying Table I.

TABLE I.—REVISION OF TABLE II, SPECIFICATIONS B 209.

Temper	Thickness, in.	Tensile Strength, psi		Yield Strength, psi		Elongation in 2 in., min, per cent
		min	max	min	max	
		ALLOY 5083				
0..............	0.051 to 2.000	40 000	50 000	18 000[h]		16
H32..........	0.051 to 0.125	45 000	54 000	34 000[h]		8
	0.126 to 0.249	45 000	54 000	34 000[h]		10
H34..........	0.051 to 0.125	50 000	59 000	39 000[h]		6
	0.126 to 0.249	50 000	59 000	39 000[h]		8
H113..........	0.188 to 2.000	44 000	56 000	31 000[h]	43 000[h]	12
		ALLOY 5456				
0..............	0.051 to 2.000	42 000	53 000	19 000[h]	30 000[h]	16
H24..........	0.051 to 0.249	51 000	61 000	39 000[h]	49 000[h]	9
H321.........	0.188 to 0.624	46 000	59 000	33 000[h]	46 000[h]	12
	0.625 to 1.250	46 000	56 000	33 000[h]	45 000[h]	12
	1.251 to 2.000	44 000	56 000	31 000[h]	43 000[h]	12

For alloy 6061-T6 in the column headed "Thickness, in.," revise the increment "2.001 to 3.000" to read "2.001 to 4.500."

For alloy Alclad 6061-T4 in the column headed "Thickness, in.," delete the data for the thickness increments "0.500 to 1.000" and "1.001 to 3.000" and replace with the thickness increment "0.500 to 3.000" and list a minimum tensile strength of 30,000, a minimum yield strength of 16,000 and a minimum elongation of 18.

Section 9.—Change the first sentence to read: "Tension test specimens for sheet or plate of alloys 1100, 1060, 5050, 5005, 5454, 5086, 5083, 5456, 5052, 5652, 5154, 5454, 3003, Alclad 3003, 3004, and Alclad 3004 shall be taken parallel to the direction of rolling; for sheet or plate of alloys 2024, Alclad 2024, Alclad 2014, 6061, Alclad 6061, 7075, and Alclad 7075, the tension test specimens shall be taken perpendicular to the direction of rolling for widths 9 in. and greater, and parallel to the direction of rolling for widths less than 9 in."

Table III.—Under the heading "Alloy," delete the ASTM designations and remove the parentheses from the AA designation.

Table IV.—Change the title to read: "Permissible variations in thickness of alloys 1100, 1060, 5050, 5005, 3003, and Alclad 3003 flat sheet, coiled sheet, and plate, plus or minus, in."

For the thickness increment 0.029 to 0.036 and width increment over 72 to 90, add the tolerance of 0.005.

For the thickness increment 0.037 to

0.045 and width increment over 72 to 90, add the tolerance of 0.006.

For the thickness increment 0.046 to 0.068 and width increment over 72 to 90, revise the tolerance from "0.007" to read "0.006."

TABLE II.—ADDITIONS TO TABLE IV, SPECIFICATION B 209 – 59 T.

Thickness, in.	Width, in.						
	18 and under	Over 18 to 36	Over 36 to 54	Over 54 to 72	Over 72 to 90	Over 90 to 102	Over 102 to 132
3.001 to 4.000	0.110	0.110	0.110	0.110	0.140	0.160	0.160
4.001 to 5.000	0.125	0.125	0.125	0.125	0.150	...	...
5.001 to 6.000	0.135	0.135	0.135	0.135	0.160	...	...

TABLE III.—REVISIONS OF TABLE V, SPECIFICATION B 209 – 59 T.

Thickness, in.	Width, in.											
	18 and under	Over 18 to 36	Over 36 to 48	Over 48 to 54	Over 54 to 60	Over 60 to 66	Over 66 to 72	Over 72 to 78	Over 78 to 84	Over 84 to 90	Over 90 to 96	Over 96 to 132
0.029 to 0.036	0.002	0.002	0.0025	0.004	0.005	0.005	0.005	0.006	0.006	...	...	...
0.037 to 0.045	0.002	0.0025	0.003	0.004	0.005	0.005	0.005	0.006	0.006	...	...	...
0.046 to 0.068	0.0025	0.003	0.004	0.005	0.006	0.006	0.006	0.007	0.007	...	...	...
0.069 to 0.076	0.003	0.003	0.004	0.005	0.006	0.006	0.006	0.007	0.007	0.012	...	...
0.077 to 0.096	0.0035	0.0035	0.004	0.005	0.006	0.006	0.006	0.007	0.007	0.012	...	...
0.097 to 0.108	0.004	0.004	0.005	0.005	0.007	0.007	0.007	0.008	0.008	0.016	0.018	0.020
0.109 to 0.125	0.0045	0.0045	0.005	0.005	0.007	0.007	0.007	0.008	0.008	0.016	0.018	0.020
0.126 to 0.140	0.0045	0.0045	0.005	0.005	0.007	0.010	0.012	0.013	0.014	0.016	0.018	0.020
0.141 to 0.172	0.006	0.006	0.008	0.008	0.009	0.012	0.014	0.015	0.016	0.017	0.019	0.023
2.251 to 2.750	0.075	0.075	0.075	0.075	0.075	0.075	0.075	0.100	0.100	0.100	0.125	0.125
2.751 to 3.000	0.090	0.090	0.090	0.090	0.090	0.090	0.090	0.120	0.120	0.120	0.150	0.150
3.001 to 4.000	0.110	0.110	0.110	0.110	0.110	0.110	0.110	0.140	0.140	0.140	0.160	0.160
4.001 to 5.000	0.125	0.125	0.125	0.125	0.125	0.125	0.125	0.150	0.150	0.150	...	...
5.001 to 6,000	0.135	0.135	0.135	0.135	0.135	0.135	0.135	0.160	0.160	0.160	...	...

For the thickness increment 2.251 to 2.750 and width increment over 102 to 132, add the tolerance of 0.125.

For thicknesses over 3.000 in., add tolerances as shown in the accompanying Table II.

Change the ASTM designation at the end of footnote *a* to read: "(ASTM Designation: E 29 – 50)."

Table V.—Revise the title to read: "Permissible variations in thickness of alloys 2024, Alclad 2024, 5454, 5086, 5083, 5456, 5052, 5652, 5154, 5254, 6061, Alclad 6061, Alclad 2014, 3004, Alclad 3004, 7075, and Alclad 7075, flat sheet, coiled sheet, and plate, plus or minus, in."

Change the tolerances for the thickness increments from 0.029 to 0.172 and for 2.251 to 3.000; also add tolerances for thickness increments over 3.000 in., as shown in the accompanying Table III.

Change the ASTM designation at the end of footnote *a* to read: "(ASTM Designation: E 29 – 50)."

Section 16 (a).—Revise the first sentence to read: "When identification marking is specified on the purchase order, all sheet and plate shall be marked with the manufacturer's name or trade mark, and the applicable alloy and temper designations, the latter to be in accordance with ASA and ASTM codification systems (ASA No. H35.1—1957 and ASTM B 296)."

Table VIII.—Correct the subheading for columns 2 through 7 to read "Permissible Variation in Width, in., for Widths Given in Inches."

New Tables.—Add new Tables XI and XII as shown in the accompanying Tables IV and V.

TABLE IV.—PERMISSIBLE VARIATION IN WIDTH AND LENGTH OF SAWED FLAT PLATE.

(New Table XI for Specification B 209 - 59 T)

Thickness, in.	Permissible Variation for Width and Length, plus and minus, in., for Widths Given in Inches			
	10 and under	Over 10 to 48	Over 48 to 84	Over 84 to 132
0.250 to 6.000....	3/32	3/16	1/4	5/16

Section 17 (b).—Revise to read: "Each package or container shall be marked with the purchase order number, size of material, specification number, alloy and temper, the latter to be in accordance with ASA and ASTM codification systems (ASA No. H35.1—1957 and ASTM B 296), gross and net weights, and name of the manufacturer."

Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes (B 210 - 59 T):

Table I.—Delete both the ASTM and AA subheadings under the column "Alloy." Delete the ASTM alloy designations in the table and footnote *a*, renumbering subsequent footnotes accordingly and revising present footnote *b* to read: "[a] These alloy designations were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No.: H35.1—1957).

Table II.—Change the ASTM designation in the Note at the top of the table to read "E 29 - 50." Delete both the ASTM and AA subheadings under the column "Alloy" and the ASTM alloy designations in the first column of the table.

TABLE V.—PERMISSIBLE VARIATION FROM FLAT.

(New Table XII, Specifications B 209)

LONGITUDINAL FLATNESS

Thickness, in.	Tolerance, in.[a]
0.250 to 6.000.....	1/4 in any 6 feet or less

TRANSVERSE FLATNESS[a]

Thickness, in.	Tolerance, in.,[a] for Indicated Width, in.		
	Up through 24	Over 24 to 48	Over 48 to 72
0.250 to 0.624.....	0.125	3/8	1/2[b]
0.625 to 1.500.....	0.090	1/4	5/16[b]
1.501 to 3.000.....	0.090	5/16	1/4[b]

SHORT CYCLE FLATNESS[c]

Thickness, in.	Tolerance, in.[d]
0.250 to 0.624.....	0.125 in any 2 feet
0.625 to 3.000.....	0.090 in any 2 feet

[a] As measured with the plate resting on a flat surface. Concave side upward, using a 6 ft long straightedge and a feeler gage, dial gage, or scale.

[b] Also applicable to any 72-in. width increment of wider plate.

[c] Short cycle flatness is the flatness over any 2-ft span in any direction.

[d] As measured with the plate resting on a flat surface, and by use of a frame with rollers mounted on 2-ft centers and a depth gage in the center.

Add mechanical property requirements for the T31 temper of 6063 tube of 0.035 to 0.094 in. wall thickness, 30,000 minimum tensile strength, 28,000 minimum yield strength, and 4 per cent minimum elongation (full section specimen).

Section 7.—Change the second

sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1060, 5050, 5052, 5652, 5154, 5254, 3003, Alclad 3003, and 3004, yield strength is not important."

Table III.—Change the subheading for column 2 to read: "Mean Diameter[a] or Pi-Tape Measurement—Alloys 1060, 2024, 5050, 5052, 5652, 5154, 5254, 6063, 6061, 6062, 3003, Alclad 3003, and 3004."

Revise the subheading for column 3 to read "Alloys 1060, 5050, 5052, 5652, 5154, 5254, 3003, Alclad 3003, and 3004."

Revise the subheading for column 4 to read: "Alloys 2024, 6063, 6061, and 6062."

Table IV.—Change the subheading for column 2 to read: "Alloys 1060, 2024, 5050, 5052, 5652, 5154, 5254, 6063, 6061, 6062, 3003, Alclad 3003, and 3004."

Change the subheading for column 3 to read: "Alloys 1060, 5050, 5052, 5652, 5154, 5254, 3003, Alclad 3003, and 3004 Round Tube Only."

Change the subheading for column 4 to read: "Alloys 2024, 6063, 6061, and 6062 Round Tube Only."

Change the ASTM designation at the end of footnote *a* to read: "(ASTM Designation: E 29 - 50)."

Table V.—Change the ASTM designation of footnote *a* to read "(ASTM Designation: E 29 - 50)."

Section 16 (a).—Revise the first sentence to read: "When identification marking is specified on the purchase order, all tubes in straightened lengths $\frac{3}{8}$ in. in diameter or across flats and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASA and ASTM codification systems (ASA No. H35.1—1957 and ASTM B 296)."

Tentative Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211 - 59 T):

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Table II.—Change the ASTM designation in the Note at the top of the table to read "E 29 - 50." Delete both the ASTM and AA subheadings under the column "Alloy" and the ASTM alloy designations in the first column of the table. Change the temper designation for Alclad 5056 from "H38" to "H393."

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1100, 1060, 5052, 5652, 5154, and 3003, yield strength is not important."

Section 13 (a).—Change to read: "(*a*) The aluminum alloy coating of Alclad 5056 wire shall have a thickness of approximately 20 per cent of the total cross-sectional area of the wire."

Section 15 (a).—Change the first sentence to read: "When identification marking is specified on the purchase order, all bars, rods, and wire in straightened lengths $\frac{3}{8}$ in. in diameter or across flats and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASA and ASTM codification systems (ASA No. H35.1—1957 and ASTM B 296)."

Section 16 (b).—Revise as indicated above for Section 17 (*b*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 - 59 T):

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Table II.—Change the ASTM designation in the Note at the top of the table to read "E 29 - 50." Delete both the ASTM and AA subheadings under the column "Alloy" and the ASTM alloy designations in the first column of the table. Revise footnote *d* alloy designation at the start of the first sentence from "CG42A" to "2024." Revise footnote *e* alloy designation at the start of the sentence from "CS41A" to "2014." Revise footnote *f* alloy designation at the start of the sentence from "CS41A" to "2014." Revise footnote *h* to read: "There are no tensile requirements for alloys 1100 and 3003 in the F temper, but the tensile strength of alloy 1100 in the F temper (as-extruded) is about 13,000 to 17,000 psi, and of alloy 3003, about 16,000 to 21,000 psi, the lower values being associated with thick or bulky sections."

Table III.—Change the ASTM designation at the end of footnote *a* to read "(ASTM Designation: E 29 - 50)."

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1100, 5454, 5086, 5083, 5456, 5154, 5254, and 3003, yield strength is not important."

New Table.—Add a new Table XII as shown in the accompanying Table VI.

Section 14 (a).—Change the first sentence to read: "When identification marking is specified on the purchase order, all bars, rods, and shapes $\frac{3}{8}$ in. in diameter or across flats and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASA and ASTM codification systems (ASA No. H35.1—1957 and ASTM B 296)."

TABLE VI.—CORNER RADII.

(New Table XII for Specification B 221 - 59 T)

Specified Radius, in.	Allowable Deviation from Specified Radius, in.
Sharp corners...............	+1/64
Under 0.188................	±1/64
0.188 and over..............	±10 per cent

Section 15 (b).—Revise as indicated above for Section 17 (*b*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 59 T):

Section 2.—In Item (*5*) revise alloy reference M1A to read "3003."

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Section 5.—In Paragraph (*a*), change alloy references M1A, GS11A, and GS11C to read 3003, 6061, and 6062, respectively. In Paragraph (*b*) revise alloy reference in the first sentence clad M1A to read "Alclad 3003."

Table II.—Change the ASTM designation in the Note at the top of the table to read "E 29 - 50." Delete both the ASTM and AA subheadings under the column "Alloy" and the ASTM alloy designations in the first column of the table.

Section 14 (a).—Change the first two lines to read: "The aluminum alloy coating of alloy Alclad 3003 tubes shall . . . ."

Section 17 (a).—Change the first sentence to read: "When identification marking is specified on the purchase order, all tubes $\frac{3}{8}$ in. in diameter and larger shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASA and ASTM codification systems (ASA No.: H35.1—1957 and ASTM B 296)."

Section 18 (b).—Revise as indicated above for Section 17 (*b*) in Tentative Specification B 209.

Appendix I.—Change the first, second, and third sentences of the second paragraph to read: "Alloys 3003 and Alclad 3003 are supplied in a strain-hardened temper to meet the specified tensile and yield strengths. Alloys 6061 and 6062 are supplied in the heat treated temper (-T4) and in the heat treated and aged temper (-T6). These alloys (6061 and 6062) are more workable in the T4 temper, and after forming work is completed, may be subsequently aged to the stronger T6 temper." Revise the third sentence of the third paragraph to read: "Alloy Alclad 3003 tubes are generally recommended in those heat exchanger services where salt or fresh cooling waters within a pH range of 5 to 8 pass through the tubes."

Tentative Specification for Aluminum-Alloy Extruded Tubes (B 235 – 59 T):

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 1060, 5454, 5456, 5052, 5652, 5154, 5254, 3003, and Alclad 3003, yield strength is not important."

Table II.—Revise as indicated above for Table II in Tentative Specification B 234.

Section 15 (a).—Change to read: "When identification marking is specified on the purchase order, all tubes shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with ASA and ASTM codification systems (ASA No.: H35.1—1957 and ASTM B 296)."

Tentative Specification for Aluminum-Alloy Pipe (B 241 – 59 T):

Table I.—Delete the line "ASTM Alloy" and the ASTM alloy designations which follow. Delete the letters "AA" from the heading "AA Alloy" and change the footnote reference from *b* to *a*. Delete footnote *a*, renumbering subsequent footnotes accordingly and revising present footnote *b* to read: "[a] The alloy designations were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No.: H35.1—1957).

Section 7.—Change the second sentence to read: "Requirements for yield strength are included for such applications as pressure vessels, but for most applications involving the use of alloys 5454, 5456, and 3003, yield strength is not important."

Table II.—Revise as indicated above for Table II in Tentative Specification B 234.

Section 14 (a).—Change the first sentence to read: "When identification marking is specified on the purchase order, all pipe shall be marked with the manufacturer's name or trade mark and the applicable alloy and temper designations, the latter to be in accordance with the ASA and ASTM codification systems (ASA No. H35.1—1957 and ASTM B 296)."

Section 15 (b).—Change to read: "Each package or container shall be marked with the purchase order number, size and schedule number of material, specification number, alloy and temper, the latter to be in accordance with

ASA and ASTM codification systems (ASA No. H35.1—1957 and ASTM B 296), gross and net weights, and the name of the manufacturer."

Tentative Specification for Aluminum-Alloy Die Forgings (B 247 - 59 T):

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Table II.—Revise as indicated above for Table II in Tentative Specification B 234.

Section 15 (b).—Revise as indicated above for Section 17 (*b*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 - 59 T):

Section 2.—In Item (*5*) change alloy reference M1A to read "3003."

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Section 5 (b).—In the first sentence change alloy reference clad M1A to read "Alclad 3003."

Table II.—Revise as indicated above for Table II in Tentative Specification B 234.

Section 15.—In Paragraph (*a*) change the first two lines to read: "15. (*a*) The aluminum alloy cladding of alloy Alclad 3003 tubes shall comprise. . . ." In Paragraph (*b*) change the first two lines to read: "(*b*) The aluminum alloy cladding used in the preparation of alloy Alclad 3003 . . . ."

Section 18 (b).—Revise as indicated above for Section 17 (*b*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Standard Structural Shapes, Rolled or Extruded (B 308 - 59 T):

Table I.—Revise as indicated above for Table I in Tentative Specification B 210. Also, for alloy 5086 correct the maximum limit for silicon to read "0.40" instead of "0.50" and correct the maximum limit for iron to read "0.50" instead of "0.40."

Table II.—Change the ASTM designation in the Note at the top of the table to read "E 29 - 50." Delete both the ASTM and AA subheadings under the column "Alloy" and the ASTM alloy designations in the first column of the table. Revise footnote *b* to read: "[b] Reheat treatment of alloy 6066 may result in properties lower than those specified."

Section 14 (b).—Revise as indicated above for Section 17 (*b*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Round Welded Tubes (B 313 - 59 T):

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Section 5 (b).—Change the first three lines to read: "(*b*) Samples for determining the chemical composition of Alclad 3004 tubing shall be taken in the same gen- . . . ."

Section 8 (a).—Change the first sentence to read as follows:

> 8. (*a*) Tension test specimens taken from the sheet of alloys 1100, 5050, 5052, 5086, 5154, 3003, and Alclad 3004 shall be taken parallel to the direction of rolling; for sheet of alloy 6061 the tension test specimens shall be taken perpendicular to the direction of rolling for widths 9 in. and greater, and parallel to the direction of rolling for widths less than 9 in.

Table II.—Revise as indicated above for Table II in Tentative Specification B 234.

Section 14 (a).—Change the first line to read: "(*a*) For Alclad 3004, the alumi- . . . ."

Section 16 (b).—Revise as indicated above for Section 17 (*b*) in Tentative Specification B 209.

Tentative Specification for Aluminum-Alloy Rivet and Cold Heading Wire and Rods (B 316 – 59 T):

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Table II.—Revise as indicated above for Table II in Tentative Specification B 234.

Table III.—Revise as indicated above for Table II in Tentative Specification B 234.

Section 14 (b).—In the fourth line delete the words "(ASTM or commercial)."

Section 15 (a).—Revise as indicated above for Section 17 (*a*) in Tentative Specification B 210.

Tentative Specification for Extruded Aluminum-Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (Bus Conductors) (B 317 – 58 T): Revise as appended hereto.[2]

Tentative Specification for Type A and Type B Aluminum-Alloy Drawn Annealed Seamless Coiled Tubes (B 318 – 59 T):

Table II.—Revise as indicated above for Table I in Tentative Specification B 210.

Table III.—Change the ASTM designation in footnote *a* to read "E 29 – 50." Delete both the ASTM and AA subheadings under the column "Alloy" and the ASTM alloy designations in the first column of the table.

Section 14 (a).—Change to read: "Each coiled tube shall be marked at intervals, not exceeding 18 in., along its length with the words "Type A" or "Type B," the manufacturer's name or trade mark, and the alloy designation. The alloy designation shall be in accordance with American Standard Alloy Designation System for Wrought Aluminum (ASA No.: H35.1—1957)."

Tentative Specification for Aluminum-Alloy Pipe for Gas and Oil Transmission and Distribution Piping Systems (B 345 – 59 T):

Section 2.—Change Item (*6*) to read: "Cladding requirements for alloys 3003 and 6061 (Section 16)."

Table I.—Revise as indicated above for Table I in Tentative Specification B 210.

Table II.—Add the following Note to the top of the table:

NOTE.—For purposes of determining conformance with this specification, each value for tensile strength and yield strength shall be rounded off to the nearest 100 psi, and each value for elongation shall be rounded off to the nearest 0.5 per cent, both in accordance with the rounding-off method of the Recommended Practices for Designating Significant Places in Specified Limiting Values (ASTM Designation: E 29 – 50).

Change the ASTM designation in the Note at the top of the table to read "E 29 – 50." Delete both the ASTM and AA subheadings under the Column "Alloy" and the ASTM alloy designations in the first column of the table.

Section 5 (b).—Revise the first sentence to read: "(*b*) Samples for determining the chemical composition of alloys Alclad 3003 and Alclad 6061 pipes shall be taken in the same general manner described in Paragraph (*a*) but must be processed in such a manner as to segregate the borings, turnings, or chips representing the core and the coating."

Table III.—Change footnote *a* to read:

[a] Based on density of 0.098 lb per cu in., the density of alloys 1060, 6061, 6062, and 6063; determined according to the equation:

$$(\text{outside diameter} - \text{wall thickness}) \times \text{wall thickness} \times 3.6945 = \text{weight in lb per linear ft.}$$

---

[2] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

For alloys 5086 and 5456, multiply by 0.980; for alloys 5050 and 5052 by 0.990; and for alloy 3003 by 1.010.

Section 16.—Change the first sentence of Paragraph (*a*) to read: "(*a*) The aluminum alloy cladding of alloys Alclad 3003 or Alclad 6061 pipes shall comprise either the inside surface of the pipe (only) or the outside surface of the pipe (only) as specified."

Change Paragraph (*b*) to read as follows:

(*b*) The aluminum alloy cladding used in the preparation of alloys Alclad 3003 and Alclad 6061 shall be fabricated in such a manner as to result in a final pipe composite having the following approximate cladding thicknesses:

Alloy and Cladding	Approximate Thickness of Cladding, per cent of specified wall thickness
Alclad 3003 (clad inside)........	10
Alclad 3003 (clad outside).......	7
Alclad 6061 (clad inside)........	10
Alclad 6061 (clad outside).......	7

Section 18 (*a*).—Revise as indicated above for Section 14 (*a*) in Tentative Specification B 241.

Section 19 (*b*).—Revise as indicated above for Section 15 (*b*) in Tentative Specification B 241.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specification for Aluminum Alloy Hardeners Used in Making Zinc-Base Die Casting Alloys (**B 327 – 58 T**) be approved for reference to letter ballot of the Society for adoption as standard with revision as indicated:

Section 6.—Add a new Paragraph (*c*) to read as follows: "(*c*) If the manufacturer has made an analysis of the material during the course of manufacture, he shall not be required to sample and analyze the finished product."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specification for Aluminum for Use in Iron and Steel Manufacture (B 37 – 59):

Section 5.—Add a new Paragraph (*c*) to read as follows: "(*c*) If the manufacturer has made an analysis of the material during the course of manufacture, he shall not be required to sample and analyze the finished product."

Standard Specification for Magnesium-Base Alloy Sheet and Plate (B 90 – 59):

Section 8.—Change to read as follows:

8. The tension test specimens shall be cut in the direction of rolling or longitudinal direction to the form and dimensions shown in Fig. 7 for material under 0.500 in. in thickness or Fig. 8 for material 0.500 in. or over of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8). If it is necessary to use specimens smaller than the standard specimens, they shall have dimensions proportional to those of Fig. 8 but not less than the following dimensions: reduced section, $\frac{1}{4}$ in. diameter × 1 in. gage length; grip ends, $\frac{3}{8}$ in. diameter; total length, 3 in. if tested with threaded or shouldered ends, or 4 in. if tested with wedge grips.

Standard Specification for Magnesium-Base Alloy Forgings (B 91 – 59):

Section 8.—Add the following sentence to the end of the section: "If it is necessary to use specimens smaller than the standard specimens, they shall have dimensions proportional to those of Fig. 8 of Methods E 8 but not less than the following dimensions: reduced section, ¼ in. diameter × 1 in. gage length; grip ends, ⅜ in. diameter; total length, 3 in. if tested with threaded or shouldered ends, or 4 in. if tested with wedge grips."

Standard Specification for Magnesium Ingot and Stick for Remelting (B 92 – 57):

Section 1.—Change to read as follows: "1. This specification covers three grades of magnesium ingot and stick for remelting: 9980A is available in ingot and stick form, and 9990A and 9995A are generally available only in ingot form."

TABLE VII.—REVISION OF TABLE I, SPECIFICATION B 92 – 57.

	9980A	9990A	9995A
Aluminum, max, per cent	...	0.003	0.01
Copper, max, per cent	0.02	...	...
Iron, max, per cent	...	0.03	0.003
Lead, max, per cent	0.01	...	...
Manganese, max, per cent	0.15	0.004	0.004
Nickel, max, per cent	0.001	0.001	0.001
Silicon, max, per cent	...	0.005	0.005
Tin, max, per cent	0.01	...	...
Titanium, max, per cent	...	...	0.01
Other impurities each, max, per cent[b]	0.05	0.01[a]	0.005[a]
Magnesium, by difference, min, per cent	99.80	99.90	99.95

[a] For nuclear applications, the cadmium and boron (high capture cross-section elements) shall be specified as follows:

	9990A	9995A
Cadmium, max, per cent	0.0001	0.00005
Boron, max, per cent	0.00007	0.00003

[b] For specific applications, other minor impurities may be required to be controlled to limiting maxima by agreement between purchaser and manufacturer.

Table I.—Retain Note 1 and Note 2 and revise to read as shown in the accompanying Table VII.

Standard Specification for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 59):

Section 8 (a).—Change to read as follows:

> 8. (*a*) Tension test specimens shall be full sections of the material or of the types shown in Fig. 7 and Fig. 8 of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8). If it is necessary to use specimens smaller than the standard specimens, they shall have dimensions proportional to those of Fig. 8 but not less than the following dimensions: reduced section, ¼ in. diameter × 1 in. gage length; grip ends, ⅜ in. diameter; total length, 3 in. if tested with threaded or shouldered ends, or 4 in. if tested with wedge grips.

TABLE VIII.—ADDITIONS TO TABLE I, SPECIFICATION B 179 – 59.

	SC51B	SG70B
Aluminum, per cent	remainder	remainder
Copper, per cent	1.0 to 1.5	0.10
Iron, per cent	0.15	0.15
Silicon, per cent	4.5 to 5.5	6.5 to 7.5
Manganese, per cent	0.10	0.10
Magnesium, per cent	0.45 to 0.6	0.25 to 0.40
Zinc, per cent	0.10	0.10
Chromium, per cent	...	...
Titanium, per cent	0.20	0.20
Nickel, per cent	...	...
Tin, per cent	...	...
Other elements, per cent:		
Each	0.05	0.05
Total	0.15	0.15

Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 59):

Section 6.—Designate as 6 (*a*) and add a new Paragraph (*b*) to read: "(*b*) If the manufacturer has made an analysis of the material during the course of manufacture, he shall not be required to sample and analyze the finished product."

Table I.—Change the iron limit for alloys G8A, S12A-B, and SG100A-B to

read "1.0 max"; revise the zinc limit for SC84A-B to read "2.9 max"; and add alloys SC51B and SG70B with the chemical requirements shown in the accompanying Table VIII.

Tables II and III.—Add alloy SC51B with the color code "Brown and Yellow" and alloy SG70B with the color code "Purple and Brown."

Standard Specification for Magnesium-Base Alloy Extruded Tubes (B 217 – 59):

Section 8.—Change to read as follows:

8. Tension test specimens from tubes 2 in. or less in outside diameter shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a specimen. For tubes over 2 in. in diameter, or in those cases when a full-section specimen cannot be used, it is permissible to use specimens of the type shown in Fig. 13 (specimen No. 1) of the Methods of Tension Testing of Metallic Materials (ASTM Designation: E 8). If it is necessary to use specimens smaller than the standard specimens, they shall have dimensions proportional to those of Fig. 8 of Methods E 8 but not less than the following dimensions: reduced section, $\frac{1}{4}$ in. diameter × 1 in. gage length; grip ends, $\frac{3}{8}$ in. diameter; total length, 3 in. if tested with threaded or shouldered ends, or 4 in. if tested with wedge grips.

APPENDIX II

ATMOSPHERIC EXPOSURE OF ALUMINUM AND MAGNESIUM SAND AND PERMANENT MOLD CASTINGS

By L. H. Adam and M. Dougherty[1]

At various periods during the past few years, the ASTM Committee B-7 on Light Metals and Alloys, has been reporting data on castings and wrought material of aluminum and magnesium alloys as outlined in the 1953[2] report to the Society. Results obtained on the sand and permanent mold cast specimens after a 6-month and 1-yr and a 3-yr exposure period were reported in 1954[3] and 1958,[4] while data recorded on the wrought material for similar exposure periods were presented in 1955[5] and 1959.[6] This program, as originally proposed by the committee, will extend through a 10-yr exposure period, and contain results from five of the ASTM exposure sites, namely, State College, Pa. (rural atmosphere), New York, N. Y. (industrial atmosphere), and Kure Beach, N. C.; Point Reyes, Calif.; and Freeport, Tex. (marine atmosphere on the East, West, and Gulf Coasts, respectively).

Information contained in this report pertains to the values recorded after a 5-yr exposure period on the aluminum and magnesium sand and permanent mold castings. Evaluations of the castings are based on the average results obtained on six specimens per alloy taken from each of the five test sites. Results are compared with those recorded on similar specimens which have been retained under control conditions for a 5-yr period.

The mechanical properties of yield strength, tensile strength, and per cent elongation in 2 in. were obtained on each specimen and the actual values are shown in Tables I through XXV. The average tensile strength and per cent elongation for each alloy at the five different exposure sites are illustrated in the bar graphs of Figs. 1, 2, and 3, together with the ASTM specifications and average control values for a 5-yr period.

Although, in general, the actual results recorded after five years' exposure were lower than those recorded after three years' exposure, observations based on respective control values showed no significant difference between per cent change after a 5-yr exposure period and that noted after a 3-yr exposure period.

---

[1] Materials Engineer and Physical Science Aide, respectively, Frankford Arsenal, Pitman Dunn Laboratories, Philadelphia, Pa.

[2] L. H. Adam, "Atmospheric Exposure of Light Metals," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 227 (1953).

[3] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 270 (1954).

[4] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 194 (1958).

[5] L. H. Adam, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 284 (1955).

[6] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 59 (1959).

The average tensile strength values for individual alloys after five years' exposure varied less than 2400 psi, when compared with the average values for a 3-yr exposure time.

With regard to per cent elongation, an example in the loss of ductility taking place during exposure is shown in values recorded on the aluminum alloy, ZG32A-T5. The per cent elongation values for six sand castings exposed at the Kure Beach test site for a 3-yr period averaged 4.5 per cent, while the values for six samples after a 5-yr exposure period at the same test site averaged 2.9 per cent.

An accumulation of data on such a variety of magnesium and aluminum alloys exposed at various sites throughout the country is being made possible through the continuing support of the various participating members of the Society. Subcommittee VIII is most grateful for their cooperation and assistance particularly in machining and testing the large quantity of specimens involved.

TABLE I. - DATA ON ALUMINUM ALLOY NO. S5B - F, SAND CASTING.

ASTM Specification Requirements[a]	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
7000 psi, min	12000 12800 12200 12500 12100 --	11400 11600 11800 11600 11500 --	11700 11300 10800 11200 12100 11000	10300 11500 10600 10600 11100 11100	13200 11200 11100 11500 11400 --	11700 11400 11600 11100 11100 12700
Average	12300	11600	11400	10900	11700	11600
TENSILE STRENGTH, PSI						
17000 psi, min	23200 23700 23200 23700 23400 --	22900 23100 22000 23600 23000 --	22600 23200 19900 22700 23400 23200	23600 22700 23000 23100 22900 23700	21900 21800 21000 20000 20900 20500	22800 22600 22900 22800 21700 22200
Average	23400	22900	22500	23200	21000	22500
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	3.8 3.8 3.9 4.0 3.8 --	3.3 3.9 2.9[b] 4.1 3.7 --	4.0 4.4 2.7[b] 4.0 4.3 4.5	3.7 3.3 3.4 3.4 3.2 3.9	2.6[b] 2.6[b] 2.5[b] 1.9[b] 2.4[b] 2.6[b]	3.7 4.0 3.7 4.3 3.5 2.6[b]
Average.	3.9	3.6	4.0	3.5	2.4[b]	3.6

[a] Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 - 58 T), 1958 Book of ASTM Standards, Part 2.

[b] Low

TABLE II. - DATA ON ALUMINUM ALLOY NO. C4A - T6, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	33100 33200 33500 34200 32800 33400	32400 32900 32900 32400 32900 32600	32300 32600 32200 32600 32000 32300	32300 31900 32200 31700 32300 32100	31000 30600 30500 30600 30000 30100	31700 31800 32200 31400 32200 32000
Average.	33400	32700	32300	32100	30500	31900
TENSILE STRENGTH, PSI						
32000 psi, min	46800 45500 45400 45600 45100 43600	43200 43400 45000 45000 40800 43600	43400 43700 42500 42900 40900 43700	41300 41500 40700 41000 40800 40800	37500 37700 37700 38600 37000 37100	40800 40200 41400 41400 40800 39300
Average.	45300	43500	42900	41000	37600	40700
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	4.0 3.9 4.0 3.7 5.3 3.5	3.5 3.9 4.3 4.1 2.6[a] 3.2	3.7 4.2 3.8 4.0 3.0 4.5	3.0 2.8[a] 2.3[a] 2.7[a] 2.9[a] 2.6[a]	1.9[a] 1.9[a] 2.1[a] 2.4[a] 2.4[a] 1.7[a]	3.0 3.1 3.1 3.4 2.8[a] 2.8[a]
Average.	4.1	3.6	3.9	2.7[a]	2.1[a]	3.0

[a] Low

TABLE III. - DATA ON ALUMINUM ALLOY NO. SC51A - T6, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	30000	34500	29800	31500	29000	31000
	31100	29200	29600	30700	28300	30800
	31000	29500	30600	30400	28000	31200
	30100	29000	29800	31300	29100	30400
	31000	33400	30400	30600	29200	31300
	31500	29100	31000	30500	29400	30700
Average. . . .	30800	30800	30200	30800	28800	30900
TENSILE STRENGTH, PSI						
32000 psi, min	36100	40900	36900	38000	35200	34700
	37500	37100	37000	37100	35400	36900
	35900	36500	37000	37100	33300	35700
	38200	37100	35700	36900	33500	35800
	37200	39800	36000	37300	34100	37100
	38400	34500	38000	35500	35200	37200
Average.	37200	37700	36800	37000	34500	36200
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	1.9[a]	1.6[a]	2.1	1.6[a]	1.9[a]	2.0
	1.6[a]	2.4	1.8[a]	1.6[a]	2.0	2.2
	1.4[a]	1.3[a]	2.0	1.2[a]	1.5[a]	1.4[a]
	2.6	2.9	1.6[a]	1.2[a]	1.3[a]	1.8[a]
	1.8[a]	2.0	1.7[a]	1.8[a]	1.6[a]	2.0
	1.8[a]	1.6[a]	2.3	1.5[a]	1.5[a]	2.5
Average.	1.9[a]	2.0	1.9[a]	1.5[a]	1.6[a]	2.0

[a] Low

TABLE IV. - DATA ON ALUMINUM ALLOY NO. SG70A - T6, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	26100	24600	27100	25100	25500	25600
	25700	26500	24900	25900	25200	26700
	25000	25900	24200	26500	26000	25600
	27400	24800	25000	25000	26400	25100
	24500	25800	25600	25700	25000	25600
	24600	24400	26400	27000	25200	26200
Average.	25600	25300	25500	25900	25700	25800
TENSILE STRENGTH, PSI						
30000 psi, min	35300	33500	35800	33500	34500	34800
	35800	35200	34600	34600	33200	33800
	36200	34400	33800	34500	35100	34300
	34400	34800	34100	35100	34400	33900
	34500	35100	33300	35200	34400	34000
	34100	34700	34100	33300	33900	34500
Average.	35100	34600	34300	34400	34300	34200
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	3.7	3.9	3.9	3.3	4.1	4.7
	4.4	3.8	5.2	4.2	3.6	2.6[a]
	5.7	3.3	3.9	3.8	3.8	4.6
	4.0	4.5	4.2	4.7	3.7	3.8
	4.6	4.0	3.8	3.8	4.4	4.3
	4.2	4.8	3.5	2.5[a]	4.0	3.0
Average.	4.4	4.1	4.1	3.7	3.9	3.9

[a] Low

TABLE V. - DATA ON ALUMINUM ALLOY NO. SC64D - T6, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	34600 35800 35200 35100 34900 35200	35200 35300 35200 34500 33700 --	33400 34300 34500 34100 34000 33200	34200 34900 34400 34800 34400 35600	33400 33800 33700 33800 32600 33900	34700 34400 34800 35300 34600 34300
Average.	35100	34800	33900	34700	33500	34700
TENSILE STRENGTH, PSI						
32000 psi, min	43400 44200 43500 44200 44200 44200	44100 44700 44500 44100 42300 --	40900 37600 42000 41600 40700 41100	42600 42000 37800 41800 41300 42800	39900 38900 38600 39900 38100 39500	42200 41200 40300 42600 41500 41300
Average.	44000	43900	40700	41400	39200	41500
ELONGATION IN 2 IN., PER CENT						
2.5 per cent, min	1.6[a] 1.9[a] 1.7[a] 1.7[a] 1.3[a] 1.7[a]	1.5[a] 1.7[a] 1.6[a] 1.8[a] 1.4[a] --	1.2[a] 0.9[a] 1.5[a] 1.3[a] 1.4[a] 1.7[a]	1.3[a] 1.5[a] 0.7[a] 1.6[a] 1.2[a] 1.6[a]	1.2[a] 1.1[a] 0.9[a] 0.9[a] 0.9[a] 0.9[a]	1.6[a] 1.6[a] 1.5[a] 1.8[a] 1.6[a] 1.7[a]
Average.	1.7[a]	1.6[a]	1.3[a]	1.3[a]	1.0[a]	1.6[a]

[a] Low

TABLE VI. - DATA ON ALUMINUM ALLOY NO. ZG61A - T5, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
25000 psi, min	33200 31900 29600 31400 31400 31200	29800 31400 28900 30300 29700 30000	30800 30900 29400 29900 30100 31300	32200 32800 29900 30100 30100 32500	29100 29900 29300 31000 29500 29300	30300 30400 29800 30100 30900 --
Average.	31500	30000	30400	31300	29700	30300
TENSILE STRENGTH, PSI						
32000 psi, min	41000 37700 37000 38500 38000 40800	37200 39800 36300 39000 37500 37600	36400 37000 36500 36700 37400 38300	38300 40400 36800 34500 36300 41100	34300 36900 35700 35600 36500 35800	35500 35400 35800 34500 36000 34200
Average.	38800	37900	37000	37900	35800	35200
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	3.0 4.2 2.7[a] 3.7 3.9 3.3	3.1 2.4[a] 3.4 3.4 3.8 4.7	3.4 2.2[a] 3.7 3.1 1.9[a] 3.8	2.6[a] 3.7 2.8[a] 2.5[a] 2.8[a] 4.2	2.8[a] 3.8 3.4 2.3[a] 3.8 4.2	2.3[a] 2.4[a] 2.4[a] 1.8[a] 2.6[a] 2.4[a]
Average.	3.5	3.5	3.0	3.1	3.4	2.3[a]

[a] Low

TABLE VII. - DATA ON ALUMINUM ALLOY NO. CS43A - F, SANDCASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
12000 psi, min	23300	23300	24000	24100	22700	22600
	23000	22200	23000	23100	23700	24400
	22400	23900	23800	24100	23300	23200
	23500	23900	23700	24200	23700	23800
	23400	22300	22700	23400	22600	23300
	24100	23900	22700	23800	--	23000
Average.	23300	23300	23300	23800	23200	23400
		TENSILE STRENGTH, PSI				
19000 psi, min	30300	31600	29300	29000	28000	25200
	29800	29100	27900	28500	26000	29100
	29400	29600	28800	28800	25900	26800
	30200	29000	29800	26900	27200	27400
	28700	27700	28600	27100	25600	27700
	29700	28900	29100	26800	--	25900
Average.	29700	29300	28900	27900	26500	27100
		ELONGATION IN 2 IN., PER CENT				
1.5 per cent, min	1.5	1.6	1.3[a]	1.3[a]	1.0[a]	1.3[a]
	1.5	1.4[a]	1.4[a]	1.2[a]	0.7[a]	1.7
	1.4[a]	1.0[a]	1.3[a]	1.0[a]	0.7[a]	1.5
	1.4[a]	1.2[a]	1.1[a]	1.0[a]	0.8[a]	1.3[a]
	1.4[a]	1.4[a]	1.4[a]	1.3[a]	1.2[a]	1.3[a]
	1.5	1.5	1.5	1.0[a]	--	1.4[a]
Average.	1.5	1.4[a]	1.3[a]	1.1[a]	0.9[a]	1.4[a]

[a] Low

TABLE VIII. - DATA ON ALUMINUM ALLOY NO. ZG32A - T5, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
17000 psi, min	32000	30000	31200	31500	29100	33300
	31300	30600	30400	31400	30800	31800
	32000	28900	30200	31900	34900	33000
	31100	29100	31100	31100	31100	30800
	32000	30800	29000	30500	28300	31400
	32100	30300	30500	31700	31100	32900
Average.	31800	30000	30400	31400	30900	32200
		TENSILE STRENGTH, PSI				
30000 psi, min	40400	36100	40000	36100	35600	38900
	38300	37700	38700	36900	37900	36100
	37800	40200	35100	38800	41600	37900
	38700	36400	39400	41800	35300	39400
	40000	39700	38100	37900	34800	36300
	41100	37700	37000	37400	37500	37600
Average.	39400	38000	38100	38200	37100	37700
		ELONGATION IN 2 IN., PER CENT				
5.0 per cent, min	4.5[a]	2.3[a]	4.9[a]	1.8[a]	3.2[a]	2.2[a]
	3.4[a]	3.4[a]	4.0[a]	2.0[a]	3.3[a]	2.3[a]
	2.4[a]	5.6	2.4[a]	2.9[a]	2.8[a]	2.6[a]
	3.2[a]	3.0[a]	3.6[a]	5.2	3.7[a]	4.1[a]
	4.0[a]	3.7[a]	4.5[a]	3.0[a]	3.4[a]	2.3[a]
	4.0[a]	3.5[a]	3.1[a]	2.7[a]	2.6[a]	2.0[a]
Average.	3.6[a]	3.6[a]	3.8[a]	2.9[a]	3.2[a]	2.6[a]

[a] Low

TABLE IX. - DATA ON ALUMINUM ALLOY NO. ZC81A - T5, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
22000 psi, min	25800	26800	25700	26300	26400	26600
	27500	27100	25900	27700	26500	25800
	26700	26300	26300	25900	26700	26100
	25400	25600	25600	27800	28000	27100
	27200	26200	26100	26200	25900	27000
	25200	26500	26500	26300	--	26800
Average.	26300	26400	26000	26700	26700	26600
TENSILE STRENGTH, PSI						
30000 psi, min	36000	34800	34400	36300	35200	35300
	38500	38000	33500	37400	34900	33900
	36800	36800	37700	35400	37100	32300
	35900	33700	34400	35400	38400	34000
	34700	38900	38400	35900	34800	33400
	34000	38000	35500	33700	--	34200
Average.	36000	36400	35700	35700	36100	33900
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	4.7	3.5	4.5	3.8	4.5	3.9
	5.0	5.4	3.4	4.1	4.2	4.3
	4.7	4.5	6.2	4.2	5.0	2.8[a]
	3.9	3.6	4.8	3.2	5.1	3.4
	2.9[a]	4.8	6.6	4.1	4.5	3.0
	3.4	5.3	4.3	2.8[a]	--	3.9
Average.	4.1	4.5	5.0	3.7	4.7	3.6

[a] Low

TABLE X. - DATA ON ALUMINUM ALLOY NO. ZG61B - T5, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	35000	34400	34200	35200	34300	35900
	35300	33800	34400	34200	33500	35300
	34100	34600	34300	35200	34300	35600
	35500	33900	34600	34100	34500	35400
	35200	34100	33900	35100	34400	36000
	35600	34200	34400	35100	33700	35100
Average.	35100	34200	34300	34800	34100	35600
TENSILE STRENGTH, PSI						
32000 psi, min	41700	41200	40400	39600	42400	41400
	40000	40600	40900	40100	37200	40100
	39000	40100	40800	41200	39000	40000
	42300	42500	40900	38200	40100	41200
	41500	38200	39600	41300	39300	42800
	41800	40800	38800	42500	39800	40300
Average.	41100	40600	40200	40500	39600	41000
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	2.4	2.6	2.4	1.4[a]	3.3	2.3
	2.3	2.6	2.8	2.4	1.9[a]	1.8[a]
	2.4	2.2	2.5	2.0	1.4[a]	2.4
	2.0	4.1	2.3	1.7[a]	2.0	2.3
	2.1	1.4[a]	1.9[a]	2.3	1.8[a]	3.3
	2.6	2.3	1.6[a]	3.0	2.4	1.8[a]
Average.	2.3	2.5	2.3	2.1	2.1	2.3

[a] Low

TABLE XI. - DATA ON ALUMINUM ALLOY NO. ZG42A - T5, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
22000 psi, min	39800	38400	37700	37500	38200	--
	39200	39100	38500	--	38300	--
	39700	37300	37000	38000	38100	35600
	40000	37800	37000	--	38000	--
	39600	38000	35800	37100	38800	35700
	40500	38500	38500	39300	38400	--
Average.	39800	38200	37400	38000	38300	35700
TENSILE STRENGTH, PSI						
33000 psi, min	45700	42900	41100	37900	40600	--
	41700	44300	41000	37600	40800	--
	42200	40000	37600	38600	41600	35800
	45500	42700	39500	36000	40400	--
	41700	40800	38500	38400	41800	37900
	45700	43100	40500	40000	39500	--
Average.	43800	42300	39700	38100	40800	36900
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	1.8[a]	2.0	1.2[a]	0.8[a]	1.6[a]	--
	1.2[a]	1.5[a]	1.2[a]	0.7[a]	1.8[a]	--
	1.3[a]	0.8[a]	1.6[a]	0.8[a]	1.8[a]	1.0[a]
	1.5[a]	1.9[a]	0.9[a]	1.0[a]	1.6[a]	--
	0.9[a]	2.0	1.0[a]	1.0[a]	1.5[a]	1.0[a]
	1.6[a]	1.8[a]	0.8[a]	0.9[a]	1.1[a]	--
Average.	1.4[a]	1.7[a]	1.1[a]	0.9[a]	1.6[a]	1.0[a]

[a] Low

TABLE XII. - DATA ON ALUMINUM ALLOY NO. S5B - F, PERMANENT MOLD.

ASTM Specification Requirements[a]	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
7000 psi, min	12200	12200	12100	11300	10800	13100
	12400	11700	13600	11600	11300	13300
	12100	12100	13000	12400	11200	12200
	12400	12100	13000	11300	10800	12500
	13300	12100	13300	11300	11000	12800
	12700	--	12900	11300	11700	12400
Average.	12500	12000	13000	11500	11100	12700
TENSILE STRENGTH, PSI						
21000 psi, min	26400	25200	26600	27200	24000	26600
	27100	27300	26600	26200	24900	24400
	26200	26600	26900	27700	24300	27300
	26900	27400	27500	27400	24700	25700
	26800	25800	26300	26700	24700	26600
	27000	--	27600	27100	25400	27300
Average.	26700	26500	26900	27100	24700	26300
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	5.4	4.4	5.7	5.1	3.4	6.6
	5.8	5.8	5.7	4.4	3.8	4.1
	5.7	5.1	5.9	6.2	4.4	6.4
	6.0	6.4	6.2	5.7	4.1	4.6
	5.4	4.8	5.6	5.5	4.3	5.8
	5.4	--	7.2	5.0	4.7	6.0
Average.	5.6	5.3	6.1	5.3	4.1	5.6

[a] Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 58 T), 1958 Book of ASTM Standards, Part 2.

TABLE XIII. - DATA ON ALUMINUM ALLOY NO. SC51A - T6, PERMANENT MOLD.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
23000 psi, min	32500	32100	29600	32800	27800	31900
	30600	32600	32200	29600	31600	31800
	29700	31300	31700	32000	32000	30000
	32800	31500	29100	30400	29100	33300
	33000	32200	31700	29400	31100	30500
	29500	--	--	31500	30200	29400
Average.	31400	31900	30900	31000	30300	31200
TENSILE STRENGTH, PSI						
37000 psi, min	44800	44200	44000	44800	37700	43100
	44700	45700	44700	43700	41000	45000
	38100	43900	42300	44500	41500	41900
	45800	45000	41500	43400	40000	45000
	48000	46400	43000	44200	42100	42900
	43200	--	--	41600	40500	42500
Average.	44100	45000	43100	43700	40500	43400
ELONGATION IN 2 IN., PER CENT						
1.5 per cent, min	3.3	3.3	4.6	3.8	2.5	2.9
	3.7	4.3	3.5	3.9	2.5	4.3
	2.1	3.5	2.8	3.6	2.4	3.3
	4.9	4.1	3.4	3.5	2.9	3.7
	4.9	4.7	3.2	3.5	2.9	3.6
	3.5	--	--	2.1	3.3	3.1
Average.	3.7	4.0	3.5	3.4	2.6	3.5

TABLE XIV. - DATA ON ALUMINUM ALLOY NO. SC64D - T61, PERMANENT MOLD.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
24000 psi, min	37600	36900	35600	37400	35100	37200
	36300	35500	36900	37000	34900	37100
	36500	37800	36900	37200	35500	40600
	37000	36200	37200	35200	34300	37700
	35900	35500	35200	36000	34900	36200
	37900	37400	35000	36300	35600	40200
Average.	36900	36600	36100	36500	35100	38200
TENSILE STRENGTH, PSI						
40000 psi, min	45500	42900	42800	44100	40000	43900
	45300	44400	43900	43000	39200	42900
	46000	44700	43500	41800	39900	45400
	44700	45100	44700	41700	38000	42000
	45400	44500	43800	42100	38700	41600
	46100	44900	43600	41000	37300	44800
Average.	45500	44400	43700	42300	39000	43400
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	1.4[a]	1.4[a]	0.8[a]	1.2[a]	0.6[a]	1.3[a]
	1.6[a]	1.8[a]	0.8[a]	0.7[a]	1.1[a]	1.2[a]
	1.8[a]	1.3[a]	0.8[a]	0.6[a]	0.8[a]	1.0[a]
	1.3[a]	1.2[a]	1.4[a]	0.7[a]	0.9[a]	0.8[a]
	1.1[a]	1.5[a]	1.1[a]	0.7[a]	0.8[a]	1.2[a]
	1.8[a]	1.1[a]	1.1[a]	0.8[a]	0.5[a]	0.9[a]
Average.	1.5[a]	1.4[a]	1.0[a]	0.8[a]	0.8[a]	1.1[a]

[a] Low

TABLE XV. - DATA ON ALUMINUM ALLOY NO. ZG32A - T5, PERMANENT MOLD.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
17000 psi, min	34800 34700 35200 35600 35200 --	32700 33300 33100 33900 32600 33000	34600 34800 34600 34200 34400 34400	34200 34500 34900 34500 34900 35100	35100 34600 34100 34500 33900 34200	35700 34900 35400 35800 35300 35400
Average.	35100	33100	34500	34700	34400	35400
TENSILE STRENGTH, PSI						
37000 psi, min	53200 52100 53700 53500 52400 --	49400 51400 48800 51900 50000 49800	52300 50700 51700 50100 51600 50600	45300 49000 48600 48500 49000 50600	52200 51100 51400 52400 50900 51400	52800 46800 46900 51500 46800 48200
Average	53000	50200	51200	48500	51600	48800
ELONGATION IN 2 IN., PER CENT						
10.0 per cent, min	11.8 11.6 12.8 10.9 10.0 --	7.9[a] 11.5 11.9 11.2 9.6[a] 9.9[a]	12.2 9.3[a] 10.5 9.4[a] 10.8 10.0	4.6[a] 7.5[a] 5.6[a] 7.1[a] 6.8[a] 7.7[a]	10.3 9.7[a] 10.4 11.6 10.6 10.4	10.2 5.5[a] 5.1[a] 8.5[a] 5.0[a] 5.6[a]
Average	11.4	10.3	10.4	6.6[a]	10.5	6.7[a]

[a] Low

TABLE XVI. - DATA ON ALUMINUM ALLOY NO. ZC60A - T5, PERMANENT MOLD.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
18000 psi, min	24000 23800 23700 24600 24400 24000	21900 23600 23500 23900 23700 23000	24400 23600 23900 24900 23500 24500	23300 24000 24000 23300 23700 24500	23000 22800 24100 23500 -- 23700	24500 25200 24100 24500 24400 --
Average.	24100	23300	24100	23800	23400	24500
TENSILE STRENGTH, PSI						
28000 psi, min	40600 39600 39200 41400 41300 39600	36300 36600 39300 38700 38800 36900	39100 40600 38000 39900 39100 39500	37700 39900 39300 33800 35800 40000	35900 36200 39300 39400 -- 39200	39800 40000 40000 40500 39000 —
Average.	40300	37800	39400	37800	38000	39900
ELONGATION IN 2 IN., PER CENT						
7.0 per cent, min	7.3 7.6 8.1 9.5 8.7 7.6	5.9[a] 6.3[a] 8.4 7.2 8.5 6.3[a]	7.2 10.4 6.9[a] 8.0 8.4 8.0	6.4[a] 6.8[a] 7.9 4.1[a] 5.0[a] 7.6	6.1[a] 6.0[a] 8.3 8.7 — 9.0	7.9 7.4 9.2 9.4 7.0 --
Average.	8.1	7.1	8.2	6.3[a]	7.6	8.2

[a] Low

TABLE XVII. - DATA ON ALUMINUM ALLOY NO. ZC81B - T5, PERMANENT MOLD

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
22000 psi, min	28000 27400 25100 24600 24000 --	23600 27600 25000 -- -- --	24900 24900 25500 24200 24100 26600	27200 24400 24800 24400 26100 27000	27500 26200 27300 26900 26300 27000	27600 25100 26700 26900 25900 26600
Average.	25800	25400	25100	25600	26900	26500
TENSILE STRENGTH, PSI						
32000 psi, min	39400 39300 31000[a] 36600 38400 --	35900 39900 34300 -- -- --	35200 34200 36300 33400 36000 30600	36000 34800 33200 35000 36800 38000	34600 35500 35600 35900 35900 35700	36300 35900 37100 36900 37100 37600
Average.	36900	36700	34300	35600	35500	36800
ELONGATION IN 2 IN., PER CENT						
4.0 per cent, min	4.8 4.4 1.6[a] 3.9[a] 5.3 --	5.1 4.9 3.1[a] -- -- --	3.8[a] 3.6[a] 4.5 2.6[a] 4.7 2.0[a]	3.2[a] 3.5[a] 2.5[a] 3.1[a] 3.9[a] 5.1	2.2[a] 3.4[a] 2.8[a] 3.4[a] 3.4[a] 3.1[a]	2.9[a] 3.9[a] 4.3 4.3 4.6 5.0
Average.	4.0	4.4	3.5[a]	3.6[a]	3.1[a]	4.2

[a] Low

TABLE XVIII. - DATA ON ALUMINUM ALLOY NO. ZG42A - T5, PERMANENT MOLD.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
25000 psi, min	43200 43600 46300 46300 43500 46400	42500 42900 42000 42200 42500 41400	43800 43000 43700 43300 43900 41000	43200 42500 43000 44100 43300 43800	43100 46000 43400 43700 43700 43300	43600 43300 44400 43700 42200 —
Average.	44900	42300	43100	43300	43900	43400
TENSILE STRENGTH, PSI						
42000 psi, min	51500 52000 52800 52500 52600 53100	49900 51600 50600 51100 50800 50000	50200 50400 51100 48600 51300 50400	48900 47200 48300 49100 48800 50300	51000 52900 52400 51400 52000 48700	45800 46600 47700 50100 46700 46700
Average.	52400	50700	50300	48800	51400	47300
ELONGATION IN 2 IN., PER CENT						
4.0 per cent, min	2.4[a] 1.8[a] 1.1[a] 1.8[a] 2.3[a] 1.5[a]	2.1[a] 1.9[a] 2.8[a] 2.4[a] 1.6[a] 1.9[a]	1.6[a] 1.9[a] 1.8[a] 2.3[a] 1.8[a] 2.2[a]	1.1[a] 1.1[a] 1.4[a] 1.1[a] 1.3[a] 1.8[a]	2.0[a] 1.5[a] 1.1[a] 1.9[a] 2.3[a] 0.8[a]	1.1[a] 1.0[a] 1.2[a] 1.7[a] 1.7[a] 1.0[a]
Average.	1.8[a]	2.1[a]	1.9[a]	1.3[a]	1.6[a]	1.3[a]

[a] Low

TABLE XIX. - DATA ON MAGNESIUM ALLOY NO. AZ63A - T4, SAND CASTING

ASTM Specification Requirements[a]	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
11000 psi, min	17400	--	16000	17300	15600	17900
	18000	16100	15700	16300	15700	19200
	16500	16800	16900	17300	15700	18100
	17400	16000	15400	17100	16400	18000
	18100	17500	16900	15700	16100	17700
	17500	17700	14800	16500	15600	17300
Average.	17500	16800	16000	16700	15900	18100
TENSILE STRENGTH, PSI						
34000 psi, min	41600	--	36300	40700	36500	40100
	41900	39900	35500	39800	40000	40800
	39500	39600	37300	38900	39100	40100
	40800	38700	36000	37800	37700	40400
	42400	40200	37500	38200	39600	40500
	41300	36500	35500	40100	38700	39900
Average.	41300	39000	36400	39300	38600	40300
ELONGATION IN 2 IN., PER CENT						
7.0 per cent, min	13.2	--	12.4	13.4	9.8	12.2
	12.7	14.3	10.6	12.2	13.4	10.8
	11.1	14.1	11.9	10.2	12.7	11.0
	12.4	12.7	12.5	10.5	10.6	12.6
	14.6	13.4	11.7	10.7	12.9	12.5
	13.4	8.3	12.0	13.1	12.2	11.9
Average.	12.9	12.6	11.9	11.7	11.9	11.8

[a] Standard Specification for Magnesium Alloy Sand Castings (B 80 - 58), 1958 Book of ASTM Standards, Part 2.

TABLE XX. - DATA ON MAGNESIUM ALLOY NO. AZ91C - F, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
10000 psi, min	17600	15300	14700	17900	17200	16900
	16700	17200	15200	17900	17400	18100
	17700	17300	14900	17500	17200	17300
	17200	17600	15000	18600	17100	16400
	18000	17900	15300	17800	17600	17000
	17000	17600	14600	17700	17300	17100
Average.	17400	17200	15000	17900	17300	17100
TENSILE STRENGTH, PSI						
18000 psi, min	25300	22900	21800	23300	23500	24000
	24400	23600	23300	23200	22600	24700
	25900	24000	23400	22500	22600	23100
	24400	24400	22400	24100	22600	23200
	26700	24100	24100	23300	23700	23800
	25600	24400	22700	23400	22500	23100
Average.	25400	23900	23000	23300	22900	23700
ELONGATION IN 2 IN., PER CENT						
No Requirement	2.3	2.3	2.1	1.4	1.9	2.4
	1.6	2.0	2.5	1.4	1.7	2.2
	2.4	2.9	2.6	1.3	1.4	2.3
	1.8	2.6	2.8	1.6	1.9	1.9
	3.0	2.6	2.1	1.9	1.8	2.4
	2.9	2.9	2.1	1.7	1.6	2.2
Average.	2.3	2.6	2.4	1.6	1.7	2.3

TABLE XXI. - DATA ON MAGNESIUM ALLOY NO. AZ92A - T6, SAND CASTING.

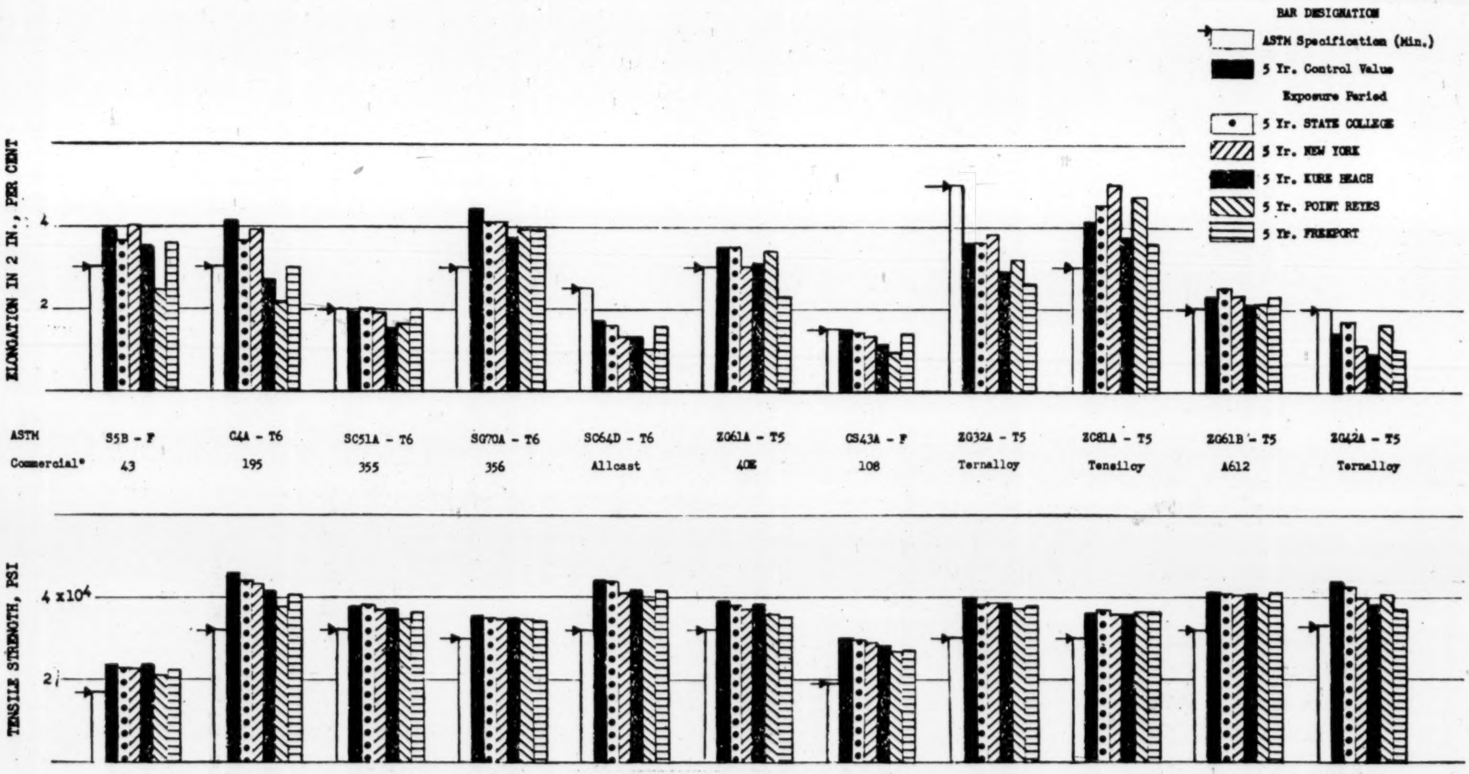
ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
18000 psi, min	23700	22400	19900	22000	21200	21900
	23400	20100	21900	21100	20000	22200
	23000	21000	17200[a]	22100	20700	20600
	22900	20500	19500	21400	20200	22100
	21800	20900	17500[a]	22800	21700	23000
	21800	20300	18600	21100	20100	21200
Average.	22800	20900	19100	21800	20700	21800
TENSILE STRENGTH, PSI						
34000 psi, min	38300	39500	34100	38800	37200	35800
	39300	38800	39000	35100	38700	36800
	41300	40500	38500	40800	39200	35100
	43000	39700	39500	39300	36800	37900
	37400	42000	38200	39100	40300	36200
	38200	37200	37900	38600	37900	36900
Average.	39600	39600	37900	38600	38400	36500
ELONGATION IN 2 IN., PER CENT						
1.0 per cent, min	2.8	3.5	2.0	3.1	2.6	3.0
	3.2	3.3	3.9	2.0	3.2	2.4
	3.6	3.7	4.9	3.4	3.3	2.3
	3.8	3.2	3.5	3.0	3.2	2.8
	2.3	5.6	3.9	2.5	3.6	3.0
	3.2	3.1	3.3	2.8	3.2	3.1
Average.	3.2	3.7	3.6	2.8	3.2	2.8

[a] Low

6

TABLE XXII. - DATA ON MAGNESIUM ALLOY NO. EK30A - F, SAND CASTING.

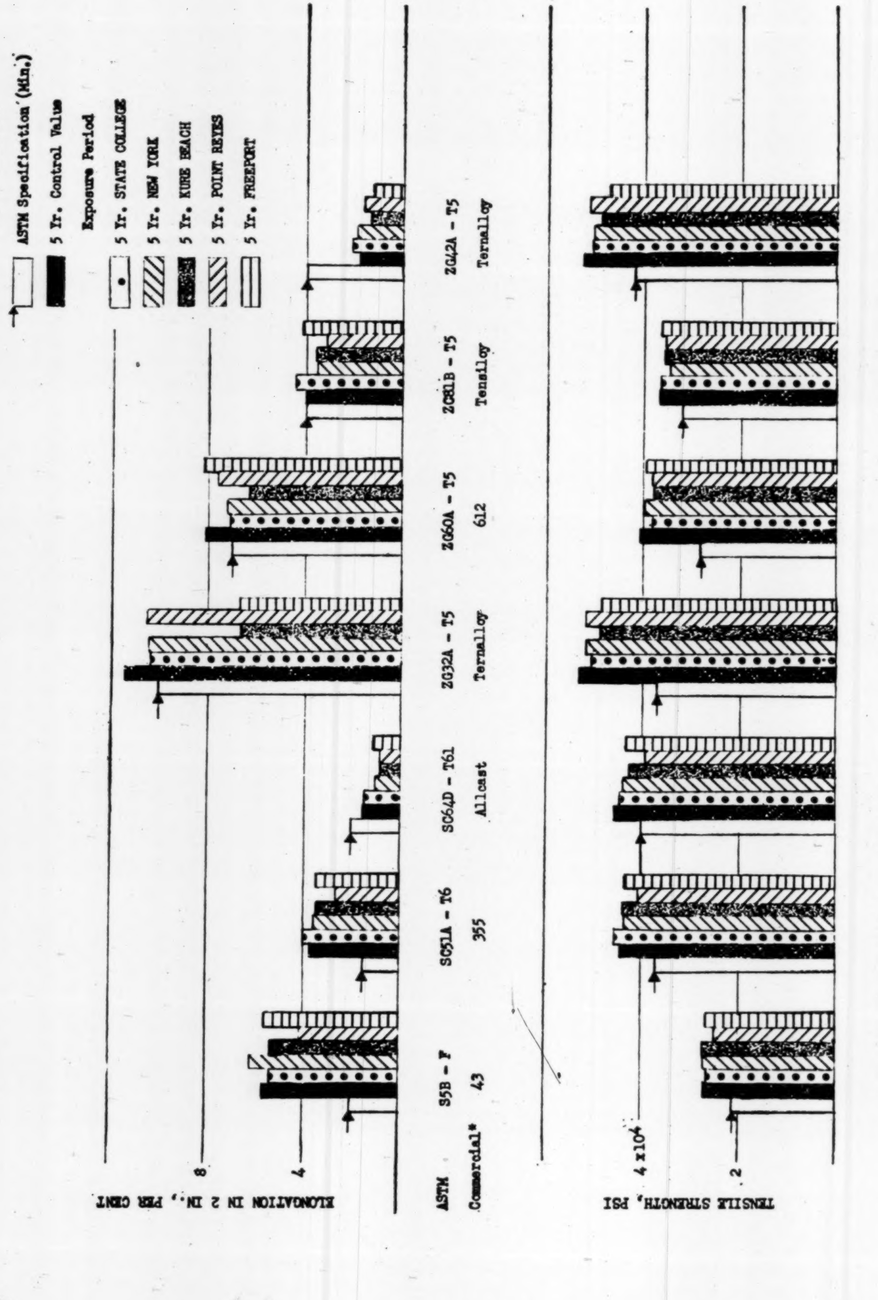
ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
No Requirements	12800	14400	11200	13800	12400	12600
	12600	13300	11600	13900	11400	11900
	13500	12800	11000	13500	12000	12200
	13700	12100	11200	13100	12700	12700
	12500	12900	11200	13600	11700	12600
	13000	13700	11500	12500	--	12200
Average.	13000	13200	11300	13400	12000	12400
TENSILE STRENGTH, PSI						
No Requirements	18300	20000	14800	18500	16500	17900
	17400	17900	18400	18000	16000	15800
	19600	17800	16200	16000	16300	16300
	18700	17400	15900	17600	17500	16900
	16800	18000	15100	18500	15000	16700
	18200	19600	17900	17100	--	16600
Average.	18200	18500	16400	17600	16300	16600
ELONGATION IN 2 IN., PER CENT						
No Requirements	1.9	3.4	1.6	1.6	2.4	2.3
	1.3	1.9	4.2	1.4	2.8	2.0
	2.3	1.9	2.8	1.0	2.2	1.7
	1.3	1.9	2.4	1.8	2.8	2.6
	1.2	2.0	1.8	2.0	2.2	1.9
	1.8	3.2	3.6	1.6	--	2.2
Average.	1.6	2.4	2.7	1.6	2.5	2.1



* As most commonly used.

Each bar represents average results on six specimens.

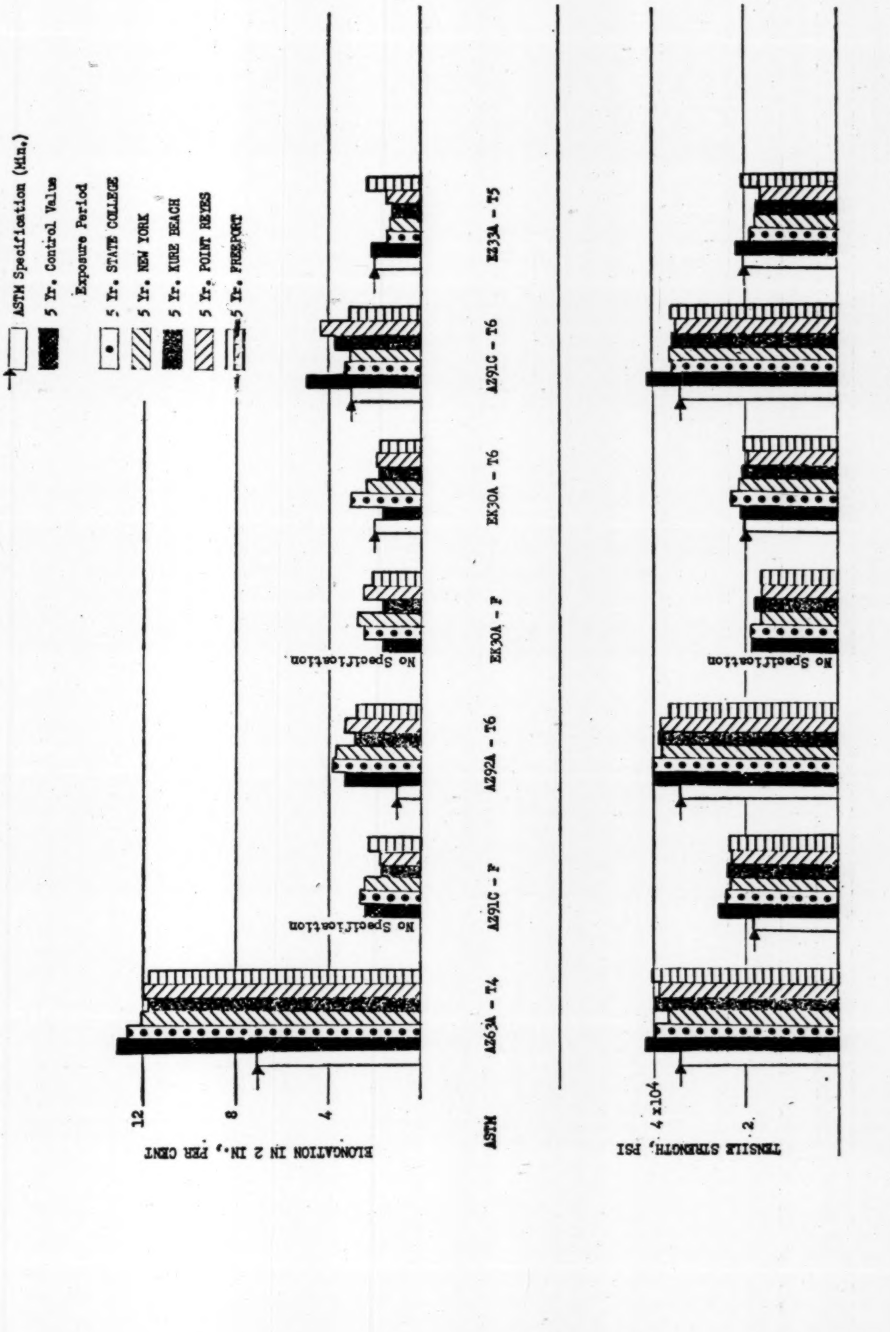
FIG. 1.—Summary of Data—Aluminum, Sandcast.



Each bar represents average results on six specimens.

* As most commonly used.

FIG. 2.—Summary of Data—Aluminum, Permanent Mold.



Each bar represents average results on six specimens.

FIG. 3.—Summary of Data—Magnesium, Sandcast.

TABLE XXIII. - DATA ON MAGNESIUM ALLOY NO. EK30A - T6, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
14000 psi, min	15200	16000	13700^a	16400	14700	14000
	16000	16900	15100	15400	14600	15100
	15300	15700	15300	14500	14600	14300
	15100	16100	15800	15200	13800^a	17600
	15400	16000	15400	15600	14000	14400
	12900^a	16200	15300	15300	14200	16100
Average.	15000	16200	15100	15400	14300	15300
TENSILE STRENGTH, PSI						
20000 psi, min	20400	22800	19000^a	21900	20500	19400^a
	22600	23100	21100	19800^a	19400^a	19900^a
	20300	21800	20700	19300^a	19200^a	19100^a
	20700	22000	21900	19800^a	18700^a	22200
	20300	22900	22100	20900	18700^a	18700^a
	20600	23500	21000	21000	19600^a	21700
Average.	20800	22700	21000	20500	19400^a	20200
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	1.4^a	2.7	1.0^a	2.4	2.4	1.5^a
	2.6	3.4	1.9^a	1.4^a	1.8^a	1.4^a
	.9^a	2.7	2.2	1.1^a	1.9^a	1.2^a
	1.9^a	2.4	2.8	1.8^a	1.8^a	3.2
	1.6^a	3.3	3.2	1.6^a	2.0	1.4^a
	1.2^a	3.6	2.4	2.4	1.7^a	2.2
Average.	1.6^a	3.0	2.3	1.8^a	1.9^a	1.8^a

[a] Low

TABLE XXIV. - DATA ON MAGNESIUM ALLOY NO. AZ91C - T6, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
16000 psi, min	22000	20600	20700	19800	20500	20900
	22000	20100	18300	20300	19800	20400
	21100	18800	22000	22100	18900	21900
	21400	20400	20600	21700	20900	23600
	21300	22100	21600	22100	15100^a	22400
	21600	20400	20000	21300	14900^a	20900
Average.	21600	20400	20500	21200	18200	21700
TENSILE STRENGTH, PSI						
34000 psi, min	40300	36600	37300	35900	35000	37500
	41700	36700	36400	33300^a	30900	35600
	42400	31800^a	36000	34200	37300	34700
	39700	37000	31800	32900^a	36100	36300
	39700	36800	37100	41300	36000	37500
	42300	34100	37800	37600	34800	35800
Average.	41000	35500	36100	35900	35000	36200
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	3.6	3.4	3.5	4.2	4.7	2.9^a
	5.5	3.4	2.9^a	3.1	3.5	2.3^a
	5.7	3.1	2.8^a	2.0^a	4.8	2.7^a
	4.8	3.7	1.3^a	2.3^a	5.2	2.7^a
	4.2	3.3	2.8^a	6.3	4.6	4.0
	5.8	2.4^a	4.6	4.4	3.7	4.2
Average.	4.9	3.2	3.0	3.7	4.4	3.1

[a] Low

TABLE XXV. - DATA OF MAGNESIUM ALLOY NO. EZ33A - T5, SAND CASTING.

ASTM Specification Requirements	5-YR CONTROL	5 - YEAR EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
14000 psi, min	16100	13400[a]	13800[a]	13800[a]	12900[a]	15800
	16400	12800[a]	13800[a]	13500[a]	13800[a]	15400
	16000	13400[a]	13800[a]	13200[a]	13100[a]	15300
	16300	13200[a]	13600[a]	13400[a]	12800[a]	15000
	15700	12900[a]	13500[a]	13400[a]	13500[a]	15600
	15600	13700[a]	13600[a]	12300[a]	13700[a]	16400
Average.	16000	13200[a]	13700[a]	13300[a]	13300[a]	15600
TENSILE STRENGTH, PSI						
20000 psi, min	19600[a]	18500[a]	17700[a]	17200[a]	15300[a]	21500
	22800	17100[a]	18000[a]	17100[a]	17400[a]	20600
	20600	16300[a]	18000[a]	16200[a]	16500[a]	20600
	22900	17100[a]	17200[a]	17300[a]	16400[a]	19300[a]
	21900	18200[a]	17600[a]	17600[a]	16600[a]	20300
	21200	22400	16400[a]	18000[a]	16300[a]	21600
Average.	21500	18300[a]	17500[a]	17200[a]	16400[a]	20700
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	1.1[a]	2.3	1.6[a]	1.3[a]	1.2[a]	2.8
	2.6	1.2[a]	1.8[a]	1.3[a]	1.0[a]	2.2
	1.4[a]	.9[a]	1.2[a]	.7[a]	1.8[a]	2.3
	2.8	1.3[a]	1.3[a]	1.0[a]	1.7[a]	1.6[a]
	2.4	1.9[a]	1.1[a]	1.2[a]	1.6[a]	2.0
	2.2	.8[a]	1.0[a]	1.8[a]	1.4[a]	2.6
Average.	2.1	1.4[a]	1.3[a]	1.2[a]	1.5[a]	2.3

[a] Low

REPORT OF COMMITTEE B-8
ON
ELECTRODEPOSITED METALLIC COATINGS AND RELATED FINISHES*

Committee B-8 on Electrodeposited Metallic Coatings held two meetings during the year: on Oct. 16, 1959, at Philadelphia, Pa., and on Feb. 5, 1960 at Chicago, Ill.

The membership of the committee totals 207, of whom 73 are classified as producers, 26 as consumers, 96 as general interest members, and 12 as consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, C. H. Sample.

Vice-Chairman, R. B. Saltonstall.

Secretary, D. M. Bigge.

Advisory Committee Member-at-large, W. H. Sefranek.

The chairman, with the concurrence of the other officers of the committee, has tentatively appointed W. S. Garwood as Membership Secretary. It is anticipated that action will be taken at the committee meeting in October 1960 to amend the By-laws to provide for this new office.

Amendment of By-laws.—The committee has submitted to letter ballot an amendment in Article VII, Section 1, of the By-laws to permit letter ballot approval of proposed standards or revisions of standards by subcommittees with approval of nine tenths of those voting instead of two thirds of its membership, as now required.

New Title and Scope.—The committee has recommended the following title and scope to define its current activities; these changes were approved by the Board of Directors in May, 1960:

Title.—Committee B-8 on Electrodeposited Metallic Coatings and Related Finishes.

Scope.—To prepare specifications for electrodeposited, anodized, and chemically reduced coatings of all kinds on all types of basis materials, subsequent treatments of such coatings, methods of testing these coatings and materials required for their production; including all pertinent investigations of the substrates, the methods of production, the properties of these deposits and their performance under conditions of use and test; excluding only specifications for metal products to which such coatings are applied.

NEW TENTATIVE

The committee recommends for publication as tentative the Recommended Practice for Preparation of Nickel for Electroplating with Nickel, as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Recommended Practice for:

Preparation of Lead and Lead Alloys for Electroplating (**B 319 – 57 T**),[2] and

Preparation of Iron Castings for Electroplating (**B 320 – 57 T**).[2]

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

[2] 1958 Book of ASTM Standards, Part 2.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Electrodeposited Coatings of Lead on Steel (**B 200 - 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Section 1.—In the first sentence change the words "to withstand corrosion" to read "to withstand atmospheric corrosion."

Footnote 3.—Add a reference to the following report on atmospheric tests: A. H. DuRose, "Atmospheric Exposure of Electroplated Lead Coatings on Steel," p. 97, *ASTM STP No. 197.*

Section 3.—Revise Note 2 of this section to read: "Copper undercoat improves the corrosion protection offered by the lead coating to the steel basis metal."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specification for:

Electrodeposited Coatings of Nickel and Chromium on Steel (**A 166 - 58 T**),[2] and
Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces (**B 201 - 55 T**).[2]

Tentative Recommended Practice for:

Cleaning Metals Prior to Electroplating (**B 322 - 58 T**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Papers, Specifications and Definitions (F. Ogburn, chairman).—Section C (W. E. Moline, chairman) prepared a Symposium on Electroless Nickel Plating, available as *ASTM STP No. 265.*

Section G (R. B. Saltonstall, chairman) is preparing a specification on multi-layer coatings of nickel and chromium on steel.

Section I (R. Ehrhardt, chairman) was established to prepare specifications for precious metal coatings. Gold and rhodium coatings will be considered first.

Subcommittee II on Performance Tests (W. L. Pinner, chairman).—Section A (B. B. Knapp, chairman) has submitted a report on Performance of Program 4 Panels to Committee B-8. Program 5, designed to study influence of the chromium deposit on durability, was approved by the subcommittee.

Section F (D. J. George, chairman) has amended its program on corrosion studies of plated aluminum to replace the bright nickel with a double-layer nickel system.

Subcommittee III on Conformance Tests (A. D. Squitero, chairman).—Section A (A. H. DuRose, chairman) has prepared a report on thickness determination of electrodeposits and one on measuring devices for thickness of zinc electrodeposited on steel. These reports are appended.[4]

Section B (A. Mendizza, chairman) is establishing a program to evaluate the clay-pack humidity (Corrodkote) and copper-accelerated salt spray (CASS) tests.

Subcommittee IV on Electroplating Practice (E. B. Saubestre, chairman).—Section N (B. B. Knapp, chairman) has prepared the Recommended Practice for Preparation of Nickel for Electroplating with Nickel referred to earlier in this report.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] See pp. 207 and 218.

Section C (B. Cohen, chairman) is studying changes to be made in Recommended Practice B 177.

Subcommittee VI on Anodic Oxidation (R. F. Hafer, chairman) appointed a task group (G. H. Kissin, chairman) to investigate the writing of a recommended practice on sulfuric acid anodizing for automotive, architectural, and electronic applications.

This report has been submitted to letter ballot of the committee, which consists of 195 voting members; 102 members returned their ballots, of whom 99 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

D. M. BIGGE,
Secretary.

APPENDIX I

REPORT OF SUBCOMMITTEE III ON CONFORMANCE TESTS—PART I: THICKNESS DETERMINATION OF ELECTRODEPOSITS

The primary objective of this work was to test the validity of Methods of Test A 219,[1] and to determine the accuracy and precision which may be expected of these methods and other methods tried. As originally stated, "It will be the responsibility of Section A to assemble information on all possible methods which might be used for measuring coating thickness and undertake such tests as are necessary to determine the accuracy and reproducibility of each promising method." The information covered in this report represents only a small part of the information needed to satisfy the above objective. Research Project 7 of the American Electroplaters' Society covered other methods, and further work is reported in Part II of this report.[2]

Coatings and Methods

Five types of coatings were used:

Group A—Approximately 0.5 mil Watts nickel on smooth steel,

Group B—Approximately 1.5 mil Watts nickel on smooth steel,

Group C—Approximately 0.5 mil Watts nickel on rough steel (rms 38 to 41 microinches),

Group D—Approximately 0.5 mil Watts nickel on smooth copper, and

Groups E-1 and E-2—Approximately 0.3 mil zinc on steel.

The thickness of the nickel coatings was determined by stripping, the magnetic reluctance gage Magne-Gage,[3] and microscopic methods.

The thickness of the group E-1 coatings was determined by the Magne-Gage, stripping, and microscope; that of group E-2 by the Magne-Gage, dropping test, and microscope.

Four laboratories, A, B, C, and D, performed the thickness determinations.

Preparation of Specimens

The original sheet for the nickel specimens was about 24 by 14.5 in. This was plated in a tank 25 in. high, 14.5 in. wide, and 24 in. long with the anodes covering one end of the tank and the sheet the other. The edges of this sheet were then cut off to give a smaller sheet 12 by 19.5 in. which was then sectioned into 104 1.5-in. squares. The rows were designated by letters and the columns by numbers. It was later decided that only 32 specimens from each sheet would be required and therefore only the center portion of this sheet was used, that is, columns 3, 4, 5, and 6 and rows *D* through *K*.

The zinc-on-steel specimens were cut from a 1.5-in. wide strip submitted by Thomas Steel Co. This strip had been cut from the center of a much wider strip. These specimens were numbered consecutively, but statistically treated

---

[1] Standard Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 – 58), 1958 Book of ASTM Standards, Part 3.

[2] See p. 218.

[3] Magne-Gage, manufactured by the American Instrument Co., Silver Spring, Md.

TABLE I.—THICKNESS AVERAGES FOR GROUP A COATINGS.

	Thickness, hundredths of a mil		
	Third Magne-Gage Method	Stripping Method	Microscopic Method
Laboratory A:			
Annealed	45.2	44.1	46.0
Not annealed	44.1	43.1	45.3
Laboratory B:			
Annealed	47.2	46.3	48.5
Not annealed	47.2	45.2	48.2
Laboratory D:			
Annealed	46.6	45.0	47.7
Not annealed	46.7	44.6	48.9
Average, laboratories A, B, and D:			
Annealed	46.3	45.1	47.4
Not annealed	46.0	44.3	47.5
Laboratory C:			
Annealed	45.0	...	45.9
Not annealed	45.8	...	50.7
Average, all laboratories:			
Annealed	46.0	...	47.0
Not annealed	46.0	...	48.3

TABLE II.—THICKNESS AVERAGES FOR GROUP B COATINGS.

	Thickness, hundredths of a mil		
	Third Magne-Gage Method	Stripping Method	Microscopic Method
Laboratory A:			
Annealed	160.6	149.1	155.8
Not annealed	164.3	148.7	150.5
Laboratory B:			
Annealed	156.8	151.1	155.3
Not annealed	159.0	152.2	154.6
Laboratory D:			
Annealed	149.9	151.3	156.4
Not annealed	152.0	149.9	156.0
Average, laboratories A, B, and D:			
Annealed	155.8	150.5	155.8
Not annealed	158.4	150.3	153.7
Laboratory C:			
Annealed	136.8	...	150.1
Not annealed	139.7	...	147.1
Average, all laboratories:			
Annealed	151.0	...	154.4
Not annealed	153.8	...	152.0

TABLE III.—THICKNESS AVERAGES FOR GROUP C COATINGS.

	Thickness, hundredths of a mil		
	Third Magne-Gage Method	Stripping Method	Microscopic Method
Laboratory A:			
Annealed	45.9	43.9	45.6
Not annealed	52.7	44.5	43.2
Laboratory B:			
Annealed	53.1	46.4	53.2
Not annealed	52.4	45.7	50.4
Laboratory D:			
Annealed	50.1	44.5	45.5
Not annealed	57.5	44.6	45.4
Average, laboratories A, B, and D:			
Annealed	49.7	44.9	48.1
Not annealed	54.2	44.9	46.3
Laboratory C:			
Annealed	48.7	...	46.7
Not annealed	50.6	...	49.0
Average, all laboratories:			
Annealed	49.4	...	47.8
Not annealed	53.3	...	47.0

TABLE IV.—THICKNESS AVERAGES FOR GROUP D COATINGS.

	Thickness, hundredths of a mil		
	Third Magne-Gage Method	Stripping Method	Microscopic Method
Laboratory A:			
Annealed	55.6	47.6	55.9
Not annealed	44.6	48.0	56.2
Laboratory B:			
Annealed	51.0	50.0	51.2
Not annealed	42.6	49.9	52.6
Laboratory D:			
Annealed	51.1	50.0	55.8
Not annealed	40.4	47.6	51.6
Average, laboratories A, B, and D:			
Annealed	52.6	49.2	54.3
Not annealed	42.5	48.5	53.5
Laboratory C:			
Annealed	48.2	...	50.6
Not annealed	37.8	...	55.2
Average, all laboratories:			
Annealed	51.5	...	53.4
Not annealed	41.4	...	53.9

in two row-column systems similar to those for nickel.

OUTLINE OF TESTING PROGRAM

1. All specimens were first measured by Magne-Gage by all four laboratories.

2. The specimens were then distributed to the four laboratories according to a latin square arrangement with the rows and columns corresponding to strips on the original large panel. Each laboratory received 8 specimens from each group.

TABLE V.—THICKNESS AVERAGES FOR GROUPS E-1 AND E-2 COATINGS.

	Thickness, hundredths of a mil				
	Third Magne-Gage Method	Third Magne-Gage Method, Burnished	Stripping Method	Dropping Test	Microscopic Method
Laboratory A	36.5	33.8	29.3	...	23.2
	37.0	...	29.6	...	23.5
	39.6	34.8	...	38.0	22.5
	37.8	...	...	39.8	25.6
Laboratory B	32.6	30.6	27.9	...	31.2
	31.8	...	27.4	...	31.6
	33.1	30.0	...	27.6	31.5
	30.6	...	...	28.8	31.8
Laboratory D	36.8	32.0	27.1	...	28.6
	33.4	...	27.1	...	28.5
	36.4	32.0	...	28.2	28.4
	32.3	...	...	28.6	28.6
Average, laboratories A, B, and D	35.3	32.2	28.1	...	27.7
	34.1	...	28.1	...	27.9
	36.4	32.3	...	31.3	27.5
	33.6	...	...	32.4	28.7
Laboratory C	30.3	30.2	...	...	21.4
	31.6	...	...	...	19.9
	30.7	29.8	...	...	26.8
	30.9	...	...	...	19.5
Average, all laboratories	34.0	31.7	...	...	26.1
	33.5	...	...	...	25.9
	35.0	31.7	...	...	27.3
	32.9	...	...	...	26.4

3. The laboratories then measured with the magnetic gage all the specimens received by them. This was called the "second Magne-Gage."

4. Half of the nickel specimens were then annealed (30 min at 400 C), and half of the zinc specimens were each burnished at two spots using the round end of a 1-lb chromium-plated weight with a 1-in. radius of curvature, and a drop of oil on the specimen.

5. All of the nickel-plated specimens were then tested by each method: 4 magnetic gage readings, 3 microscopic readings and 1 stripping evaluation (see appended methods[4]). Since there were 4 methods for the zinc coating, the odd numbered specimens, group E-1 (considered in a row-column latin square arrangement), were tested by the magnetic gage, microscopic, and stripping

---

[4] See p. 215.

methods, and the even numbered specimens, group E-2, by magnetic gage, microscopic, and dropping tests (see appended methods[4]). The magnetic gage values obtained at this stage were called "third Magne-Gage."

6. The data were then treated statistically. Since there were enough data in the third Magne-Gage stage, the data from the "second Magne-Gage" were not used, and those of the "first Magne-Gage," very little. As will be noticed, some data were not available from laboratory C.

RESULTS

In Tables I through V average results are tabulated for the coatings, methods, and laboratories.

All tabulated thickness values are given in hundredths of a mil; for example, 150.0 equals 1.50 mils. In these tables the values given are the grand averages for the four individual averages of each of the four specimens of one kind determined by one laboratory. By "individual averages" are meant the following:

For the Magnetic Gage.—The average of 4 thickness determinations on one specimen, each determination being the estimate of the average of several "flips" (see appended methods[4]). For burnished zinc it is the average of only 2 readings on one specimen.

For Stripping.—One determination on one specimen.

For Microscopic Method.—The average of 3 readings along one edge of one specimen.

For Dropping Test.—The average of 3 to 7 determinations on one specimen.

In those cases where stripping, dropping test, and microscopic results are given for burnished zinc, it should be understood that these determinations were made on the unburnished areas, not on the burnished areas.

Tables VI and VII show some differences between averages, due to methods and annealing.

TABLE VI.—DIFFERENCE BETWEEN METHODS.

(Values are differences between method averages over all laboratories)

Coating	Differences in thickness, hundredths of a mil		
	Stripping and Magne-Gage Methods	Magne-Gage and Microscopic Methods	Stripping and Microscopic Methods
Group A:			
Annealed	−0.9	−1.0	−1.9
Not annealed	−1.7	−2.3	−4.0
Group B:			
Annealed	−0.5	−3.4	−3.9
Not annealed	−3.5	1.8	−1.7
Group C:			
Annealed	−4.5	1.6	−2.9
Not annealed	−8.4	6.3	−2.1
Group D:			
Annealed	−2.3	−1.9	−4.2
Not annealed	7.1	−12.5	−5.4
Group E-1:			
Burnished	−3.6	5.7	
Not burnished	−5.6	7.7	2.1
Group E-2:			
Burnished	0.2[a]	4.8	
Not burnished	−2.1[a]	7.1	5.0[a]

[a] Drop test replaced stripping test.

TABLE VII.—EFFECT OF ANNEALING ON DIFFERENCE IN THICKNESS.

(Values are differences between average for laboratories on annealed minus similar average for unannealed)

Method	Difference in thickness, hundredths of a mil			
	Group A	Group B	Group C	Group D
Magne-Gage	0.0	−2.8	−3.9	10.1
Stripping	0.8	0.2	0.0	0.7
Microscopic	−1.3	2.4	0.8	−0.5

The analysis of variance tests for row, column, and laboratory differences are summarized in Tables VIII and IX, and

TABLE VIII.—STANDARD DEVIATIONS ASSOCIATED WITH "INDIVIDUAL AVERAGES."

Coating	Method		
	Magne-Gage	Stripping	Microscopic
Group A:			
Not annealed	0.92	0.90	4.39 (1.34)[a]
Annealed	1.21	0.60	2.06
Group B:			
Not annealed	1.40	1.72	1.22
Annealed	1.26	1.05	5.16 (4.58)[a]
Group C:			
Not annealed	2.49	1.10	2.16
Annealed	2.42	0.30	3.38
Group D:			
Not annealed	0.54	0.66	2.55
Annealed	0.78	0.74	1.26
Group E-1:			
Not burnished	1.79	0.72	1.62
Burnished	1.03	0.82	1.81
Group E-2:			
Not burnished	1.82	2.06[b]	2.66
Burnished	1.16	1.89[b]	2.32

[a] Value of standard deviation after omission of most anomalous reading.

[b] Dropping test.

TABLE IX.—SUMMARY OF RESULTS OF ANALYSIS OF VARIANCE TEST OF SIGNIFICANCE OF DIFFERENCE BETWEEN ROWS AND COLUMNS OF A PANEL AND BETWEEN LABORATORIES.

Coating	Method		
	Magne-Gage	Stripping	Microscopic
Group A:			
Not annealed	*R*, *L*[a]	*R*, *L*[a]	*R*, *L*[a]
Annealed	*R*	*R*, *L*	*R*
Group B:			
Not annealed	*R*, *L*	...	*C*, *R*, *L*
Annealed	*R*, *L*	*R*, *L*	...
Group C:			
Not annealed	*R*, *L*	*R*	*L*
Annealed	*R*, *L*	*R*, *L*	...
Group D:			
Not annealed	*L*	*L*	...
Annealed	*L*	*L*	*L*
Group E-1:			
Not burnished	*L*	*L*	*L*
Burnished	*L*	*L*	*L*
Group E-2:			
Not burnished	*L*	*L*[b]	*L*
Burnished	*L*	*L*[b]	*L*

[a] R = significant differences among rows (F test at 5 per cent level); C = significant differences among columns (F test at 5 per cent level); and L = significant differences among laboratories (F test at 5 per cent level).

[b] Dropping test.

TABLE X.—SAMPLE ANALYSIS OF VARIANCE COMPUTATIONS: DATA FOR GROUP A NOT ANNEALED, MAGNE-GAGE METHOD.

Row	Column 3	Column 4	Column 5	Column 6	Row Average	Laboratory Averages
E	43.9 (C)	44.0 (B)	41.0 (A)	44.5 (D)	43.3	A: 44.1
F	45.0 (D)	43.0 (A)	44.7 (B)	44.2 (C)	44.2	B: 47.2
H	46.0 (A)	46.6 (D)	47.0 (C)	49.0 (B)	47.2	C: 45.8
K	50.9 (B)	48.3 (C)	50.7 (D)	46.4 (A)	49.1	D: 46.7
Column averages	46.4	45.5	45.8	46.0	46.0	

Analysis of Variance	Degree of Freedom	Mean Square	F[a]
Between columns	3	$C = 0.6550$	$C/E = 0.79$
Between rows	3	$R = 27.9217$	$R/E = 35.57$
Between laboratories	3	$L = 7.2467$	$L/E = 8.71$
Error	6	$E = 0.8317$	
Total	15		

NOTE.—Standard deviation for method = $\sqrt{0.8317} = 0.92 = s_w$.

[a] If computed value of F exceeds $F_{(05,\ n_1=3,\ n_2=6)} = 4.76$, the factor under consideration is said to have significant differences among the different levels of that factor.

computations for a typical analysis of variance are shown in Table X.[5]

Differences between values on the same specimen by a laboratory should reflect only differences between methods. The sign test was used to test the significance of the difference between methods to avoid assumptions about equality of variance by the two methods. The results are given in Table XI.

TABLE XI.—RESULTS OF TEST OF SIGNIFICANCE OF DIFFERENCE BETWEEN METHODS.

Coating	Method		
	Magne-Gage, Stripping	Magne-Gage, Microscopic	Stripping, Microscopic
Group A:			
Not annealed	1–12D[a]	2–16D	0–12D
Annealed	1–12D	4–16	1–12D
Group B:			
Not annealed	0–11D	7–15	0–11D
Annealed	3–12	5–16	2–12D
Group C:			
Not annealed	0–12D	2–16D	3–12
Annealed	2–12D	4–16	2–12D
Group D:			
Not annealed	0–12D	0–16D	0–12D
Annealed	0–12D	4–16	0–12D
Group E-1:			
Not burnished	0–12D	2–16D	4–12
Burnished	0–12D	4–16	4–12
Group E-2:			
Not burnished	3–12[b]	3–16D	[illegible]–12[b]
Burnished	4–12[b]	4–16	6 [illegible] 2[b]

[a] D indicates methods are statistically significantly different at 5 per cent level of significance. The differences between paired values by any two methods were inspected and the number of differences of like sign counted. The table gives the number of like signs for the sign occurring least frequently, together with the total number of signs involved.

[b] Dropping test replaced by stripping test.

The within-laboratory variance and between-laboratory variance are related by the equation:

$$s_b^2 + \frac{s_w^2}{n} = \frac{L}{n}$$

where:

s_b^2 = the between-laboratory variance,

s_w^2 = the within-laboratory variance (error of Table X), and

L = the mean square for laboratories (see Table X).

In other words the within-laboratory variance refers to the variance of repetitive measurements that would be reported by single laboratories. Between-laboratory variance refers to variance between the measurements made by each laboratory on the same specimen, after subtracting the within-laboratory variance. These values are given in Table XII.

The magnetic gage measurements

---

[5] For the stripping test where only three laboratories reported, the analysis of the reduced latin square (an incomplete block design) was made for group A. When it developed that the columns showed significant differences only once in 24 times for the Magne-Gage and microscopic methods, and also that differences were not significant in the incomplete block analysis, it was assumed that column differences could be ignored and the analysis was carried out on the 4 block × 3 laboratory design (a 2-way classification design).

TABLE XII.—BETWEEN- AND WITHIN-LABORATORY COMPONENTS OF VARIANCE.

Coating	Magne-Gage Method		Microscopic Method[a]		Stripping and Dropping Method[b]	
	Within-Laboratory Variance, s_w^2	Between-Laboratory Variance, s_b^2	Within-Laboratory Variance, s_w^2	Between-Laboratory Variance, s_b^2	Within-Laboratory Variance, s_w^2	Between-Laboratory Variance, s_b^2
Group A:						
Annealed	1.4531	0.7606	4.2465	0.6110	0.3744	1.4296
Not annealed	0.8317	1.6038	19.2808	0.2527	0.8033	1.3533
Group B:						
Annealed	1.5915	109.5819	26.7048	1.8727	1.0997	1.5604
Not annealed	1.9688	110.1942	1.5060	14.9706	2.9603	3.4019
Group C:						
Annealed	5.8715	7.4885	11.4490	10.4708	0.0914	1.1401
Not annealed	6.2025	7.0606	4.6733	9.8296	1.2180	0
Group D:						
Annealed	0.6156	9.3617	1.5908	7.6694	0.5433	2.2223
Not annealed	0.2890	8.5642	6.4917	3.1683	0.4356	1.5025
Group E-1:						
Burnished	1.0656[a]	4.7625	3.2865	20.1614	0.6758	1.4756
Not burnished	2.0081	9.3148	2.6317	26.0671	0.5192	2.3221
Group E-2:						
Burnished	1.3423[c]	5.0775	5.3640	12.8308	3.5808[d]	44.1623[d]
Not burnished	1.2742	14.7919	7.1017	25.9240	4.3575[d]	52.4577[d]
Not burnished	3.2967[c]	10.3638				

[a] Variance for an average of 3 measurements.

[b] Variance for one stripping measurement and for an average of 3 to 7 dropping test measurements.

[c] Variance for average of 2 measurements. Each of the other Magne-Gage values is for an average of 4 measurements.

[d] Dropping test.

TABLE XIII.—STANDARD DEVIATION OF AVERAGE OF FOUR READINGS PER SPECIMEN BY MAGNE-GAGE METHOD.

Coating	First Magne-Gage Reading—from Analysis of Variance	Third Magne-Gage Reading	
		From Analysis of Variance (from Table VIII)	Based on Range of Repeat Measurements on a Specimen
Group A:			
Annealed	1.50	1.21	0.38
Not annealed	1.18	0.92	0.46
Group B:			
Annealed	...	1.26	0.91
Not annealed	...	1.40	0.69
Group C:			
Annealed	2.89	2.42	1.31
Not annealed	2.52	2.49	1.59
Group D:			
Annealed	...	0.78	0.28
Not annealed	...	0.54	0.36
Group E-1:			
Burnished[a]	...	1.03	0.55
Not burnished[a]	...	1.79	2.07, 1.53
Group E-2:			
Burnished[a]	...	1.16	1.15
Not burnished	...	1.82	1.43, 1.36

[a] Based on two measurements per specimen.

made at four locations on each specimen show closer agreement than would be expected from the standard deviations given in Table VIII, which are based on an analysis of the averages for each specimen. Table XIII gives the standard deviations from Table VIII together with estimates of the same standard deviations based on average range of readings taken within a specimen. A similar comparison for the microscopic method is given in Table XIV.

TABLE XIV.—STANDARD DEVIATION OF AVERAGE OF THREE READINGS BY MICROSCOPIC METHOD.

Coating	From Analysis of Variance (from Table X)	Based on Range of Repeat Measurements on a Specimen
Group A:		
Annealed	2.06	0.86
Not annealed	4.39 (1.34)	1.16
Group B:		
Annealed	5.16 (4.58)	1.13
Not annealed	1.22	1.71
Group C:		
Annealed	3.38	0.94
Not annealed	2.16	1.27
Group D:		
Annealed	1.26	0.64
Not annealed	2.55	1.36
Group E-1:		
Burnished	1.81	0.88
Not burnished	1.62	0.70
Group E-2:		
Burnished	2.32	0.80
Not burnished	2.66	1.07

The closer agreement within specimens is probably due to variations in the method with time as measurements are made on different specimens. The readings within one specimen are undoubtedly made within a relatively short time interval so that any temporal effect is minimized.

The analysis of the data from the first Magne-Gage readings supports this conjecture. Table XV shows the analysis of variance for the groups A and C specimens. The standard deviation for error is in close agreement with the values for the third Magne-Gage readings determined by analysis of variance.

The analysis of variance in Table XV is enlightening on another point. Inherent in the analysis of the latin square design is the assumption that the row and column effects are fixed additive constants—that is, that the row × column ($R \times C$) interaction is negligible. In all four cases no significant $R \times C$ interaction is found, and one would accept the hypothesis that additive row and column effect explains adequately the inhomogeneity, if any, in the panels. This implies that the error terms on which the standard deviations are based are not inflated by lack of uniformity among specimens.

TABLE XV.—ANALYSIS OF VARIANCE FOR DATA ON FIRST MAGNE-GAGE READING.

Coating	Degrees of Freedom	Mean Square
Group A, annealed:		
Rows of panel	3	2.7814
Columns of panel	3	81.8668
$R \times C^a$	9	2.8165
Laboratories	3	102.4181
Error	45	2.2443 = $(1.498)^2$
Group A, not annealed:		
Rows of panel	3	4.7910
Columns of panel	3	117.2747
$R \times C$	9	1.5468
Laboratories	3	73.2252
Error	45	1.3925 = $(1.180)^2$
Group C, annealed:		
Rows of panel	3	6.7210
Columns of panel	3	69.2814
$R \times C$	9	4.9022
Laboratories	3	21.1743
Error	45	7.8888 = $(2.887)^2$
Group C, not annealed:		
Rows of panel	3	6.7560
Columns of panel	3	34.4081
$R \times C$	9	11.1604
Laboratories	3	29.9389
Error	45	6.3638 = $(2.523)^2$

[a] $R \times C$ indicates row × column interaction.

CONCLUSIONS

1. Laboratories differ significantly on all methods for almost all coatings.

2. For all coatings except possibly zinc, the differences between methods are almost always significantly different from zero. For most nickel coatings on steel, the Magne-Gage and microscopic methods give significantly higher results than does the stripping test. The microscopic method gives higher results for nickel on copper than does the stripping method. The Magne-Gage method gives higher results on annealed coatings and lower results on unannealed coatings of nickel on copper than does the stripping method. Because in most cases both the Magne-Gage and microscopic methods give higher results than the stripping method for nickel deposits, there is better agreement between the Magne-Gage and microscopic methods.

The conclusions here regarding the microscopic method strictly apply only to this testing program. It is possible that a different polishing and etching technique would give different results. Where other comparisons have been made, however, the microscopic method for nickel thickness usually gives high results.

3. The Magne-Gage method for zinc on steel gives significantly higher results than the stripping method.

4. There is no consistent plus or minus difference between the microscopic and stripping methods for zinc on steel.

5. The dropping test on zinc shows no significant difference from either the Magne-Gage or microscopic methods.

6. Burnishing the zinc coatings caused a significant lowering of the Magne-Gage result, approaching better the stripping result.

7. Annealing of nickel on copper causes a significantly different Magne-Gage result, but annealing of nickel on steel does not result in a significant difference.

8. In all cases the precision for the stripping method is better than for the other methods. In addition to this, intuitively it would seem that the stripping method would give more accurate results. This is why in the previous conclusions the results of the other methods have been compared with those of the stripping method.

9. In most cases the precision for the Magne-Gage is better than that for the microscopic method.

10. For zinc, the precision for the Magne-Gage is better over burnished areas than over unburnished areas.

11. There is no consistent difference in the relative magnitude of the within-laboratory and between-laboratory variances.

12. For both the Magne-Gage and microscopic methods there is better precision for measurements made within a short period of time than for those over a longer period of time.

Acknowledgment:

Personnel in the following institutions or companies contributed to this work: National Bureau of Standards, Crane Co., Doehler Jarvis Co., Electric Auto Lite Corp., Harshaw Chemical Co., Pennsylvania State University (Dr. H. Read), Sam Tour and Co., and Sunbeam Corp.

THICKNESS DETERMINATION METHODS

Magne-Gage:

In addition to the general directions in Methods A 219, the following specific instructions were given and used.

Repeated dial readings at one point were taken until five readings of close proximity were obtained (obvious mavericks were ignored). The average of the five dial readings was then estimated and this average used to determine the thickness for this point from the curves.

The thickness was then determined at each of the four corners about $\frac{3}{8}$ in. from each edge at each corner. A grand average for the specimen was the mean of the four corner readings. For the burnished zinc specimens, only two Magne-Gage thickness determinations were made, one on each of the two burnished areas.

For the first Magne-Gage determinations a common set of thickness standards was circulated with the specimens. The specimens for each group were examined in a random order which was different for each laboratory.

For the "second and third Magne-Gage" each laboratory used its own thickness standards (supplied with the instrument) to calibrate the instrument, and curves were drawn from this data.

Dropping Test:

Each laboratory followed its own interpretation of the instructions in Methods A 219.

Microscopic Determination:

Other than that three thickness readings were to be made on a $\frac{1}{4}$-in. strip cut from the bottom of the specimen, specific instructions were not given. The following procedures were used:

Laboratory A.—The $\frac{1}{4}$-in. strip was bent and mounted. The nickel-plated specimens were polished with Nos. 1, 0, 00, and 000 metallographic emery paper. The zinc-coated specimens were wet polished on Struers wet polisher. All specimens were then fine polished with Diamet-Hyprez (6 μ) abrasive with kerosine lubricant. The nickel on steel was etched for 10 sec in 5 per cent nitric acid in alcohol. The nickel on copper was etched in 1 to 1 ammonium hydroxide - 3 per cent hydrogen peroxide. The zinc specimens were not etched. Thickness was determined at 500×.

Laboratory B.—Each $\frac{1}{4}$-in. strip was cut into three pieces and these three pieces mounted in bakelite. All mounts were rough polished with Nos. 180 and 320 grit belts parallel to the plate. All were then polished with 0, 00, 000, and 0000 emery polishing paper, beginning perpendicular to the strips and ending parallel to the strips. They were then buffed with a cloth wheel parallel to the strips, using lime buffing compound. Etching was according to Methods A 219 except that the zinc-coated steel was etched in an ammonium hydroxide - peroxide solution.

Laboratory C.—All strips were cyanide copper flashed and then heavily copper plated. Four to eight specimens were interleaved with copper foil held in steel clamps. They were polished using Nos. 1, 0, 00, and 000 metallographic emery paper. For the nickel-coated specimens, the direction of polishing was alternated between 45 and 135 deg. The final polishing was on Buehler's Microcloth at 200 rpm using Linde's type A abrasive.

Nickel on steel was etched in 2 per cent nitric acid in ethyl alcohol, nickel on copper in an ammonium hydroxide - peroxide solution; and zinc in a solution of 200 g of chromic acid and 15 g of sodium sulfate per liter.

Laboratory D.—The strips were held in a steel clamp and separated by copper spacers. Several cuts were taken across the mount with the shaper. All mounts were then polished on Nos. 1, 0, 00, and 000 metallographic paper, alternately perpendicular and parallel to strips. The nickel-plated specimens were then fine polished with No. 400 grit silicon carbide, No. 600 grit aluminum oxide on billiard cloth, and finally on Gamal levigated alumina at 250 rpm. The direction of polishing was 45 and 135 deg to the plated surface in the direction of hardest metal to the softest. Owing to galvanic action, the above fine polishing was not used on the zinc specimens. These were first lapped on a wax wheel impregnated with 25-μ emery, then on a Microcloth wheel with 4- to 8-μ diamond paste in kerosine and finally on a similar wheel with 1- to 2-μ diamond paste. All were etched according to Methods A 219.

Stripping:

There are no instructions for this method in Methods A 219 and no specific instructions were given. Several alternative methods were suggested, however. In general, the methods involved weighing the specimen, dissolving the deposit, and weighing. For the nickel on copper, the copper may be dissolved and the deposit weighed. The densities used for nickel and zinc were 8.90 and 7.13 respectively. The following are some of the methods used by the laboratories on these specimens:

Laboratory A.—Nickel on steel was stripped in a 1 to 1 mixture of concentrated and fuming nitric acid with air agitation. Copper was stripped from the nickel-on-copper specimens in a solution of 480 g chromic acid and 22 ml sulfuric acid per liter, at 100 F. The deposit on the back of the zinc-coated specimens was removed by polishing and the zinc on the face by immersion in equal parts by volume of ammonium hydroxide and 3 per cent hydrogen peroxide. The areas were determined with a planimeter.

Laboratory B.—The edges and backs of the specimens were polished. Dimensions were taken with calipers at three points in each direction. Nickel on steel was dissolved by suspending the specimens with iron hooks in the 1 to 1 mixture of concentrated and fuming nitric acid at room temperature. The 1.5-mil deposits required 12 to 16 hr. Copper was dissolved from nickel and zinc from steel by the same methods used by laboratory A.

Laboratory C.—Red fuming nitric acid with air agitation was used to dissolve nickel on steel. Copper was dissolved from nickel by the same method as in laboratories A and B. Zinc was dissolved in 1 to 1 hydrochloric acid, and rinsed quickly.

Laboratory D.—The specimens were burred and the edges smoothed with a pillar file. With vernier calipers each specimen was measured in three places in each direction. A No. 60 hole was drilled in each nickel-plated specimen and corrections made for area. The specimens were weighed to 0.0001 g and the nickel-on-steel specimens suspended on iron wire hooks in red fuming nitric acid with air agitation. The 0.5-mil deposits required 30 min to dissolve. New acid was used every 2 hr. The specimens were removed from the nitric acid and immediately dipped into a 10 per cent chromic acid solution, rinsed in water, then in alcohol, and then dried. The copper was stripped from the nickel deposit in a solution of 500 g chromic acid and 50 g sulfuric acid per liter. The fronts of the zinc-on-steel specimens were first protected with dental inlay wax and the zinc on the back stripped in 1 to 1 hydrochloric acid. The wax was then removed, the specimen cleaned and then weighed. The zinc was then dissolved as above, the specimen rinsed quickly, dipped in alcohol, dried, and weighed.

APPENDIX II

REPORT OF SUBCOMMITTEE III ON CONFORMANCE TESTS—PART II: EVALUATION OF MEASURING DEVICES FOR THICKNESS OF ZINC ELECTRODEPOSITED ON STEEL

Section A of Subcommittee III on Conformance Tests of Committee B-8 on Electrodeposited Metallic Coatings has responsibility for assembling information and undertaking tests which pertain to accuracy of methods proposed for thickness determination of electrodeposited metallic coatings. The report which follows concerns an experiment designed to evaluate thickness from four instruments (Magne-Gage, Dermitron, Boonton gage, and Kocour gage)[1] when they were used to measure zinc electrodeposited on steel. The coating thickness ranged from a minimum of about 0.1 mil to a maximum of about 0.7 mil.

PREPARATION OF TEST SPECIMENS

The test panels were prepared by two companies under their own conditions for obtaining good uniformity of thickness for the preparation of thickness standards. Three panels of each nominal thickness 0.1, 0.2, and 0.4 mil were electroplated in the form of disks 2½ in. in diameter. They were plated in a horizontal position in Lucite cylindrical cells about 2½ in. in diameter and 6 in. high. The disks were mounted at the bottom of the cell and the area plated was a circle 2¼ in. in diameter. The anodes were platinum disks ¾ in. in diameter which had been sealed into the ends of glass tubing. The anode to cathode distance was about 4½ in. The plating solution used was of conventional composition. The panels were plated at room temperature without agitation. Each panel was weighed on an analytical balance before and after plating. Thickness was calculated from the weight of deposit, using 7.14 g per cu cm as the density of zinc. These thickness results were also checked by a stripping test, and due to an obvious gross error in the case of one panel the weight gain results were used as a basis for comparison of thickness results for this group of panels.

The principle of the plating cell arrangement is a modification of Mohler and Schaefers' work[2] in which it was shown that if insulating walls are extended from the cathode to a distance of at least one and one half times the width of the cathode, the deposit is substantially uniform. After determining that the panels were of uniform thickness by the Magne-Gage they were carefully cut into four equal pieces by a milling machine. One section was reserved for a chemical stripping test and the other three sections distributed to the three laboratories. The other two thicknesses of zinc, nominal 0.3 and 0.7 mil, were plated as square cathodes 6 by 6 in. in a rectangular box with 10½ in. between anode and cathode. The current density used was 20 amp per sq ft, with periodic reverse current. As an added precaution the cathode was rotated 90 deg at each one quarter of the total plating period. A 1-in. strip was cut off the outside edges, leaving a 4 by 4 in. test panel. Twelve test specimens 1 by 1¼ in. were cut from these panels after it was determined by the Magne-Gage that the plating was of uniform thickness. These specimens were identified by code letters and 3 were used in determining the weight loss due to chemical stripping. The average of three tests was used for comparison with the results of thickness measurement tests. The remaining 9 specimens were distributed on a random basis to the three cooperating laboratories for thickness measurements.

---

[1] Magne-Gage, manufactured by American Instrument Co., Silver Spring, Md.; Dermitron, Unit Process Assemblies Inc., New York, N.Y.; Boonton gage, Boonton Radio Corp., Boonton, N.J.; Kocour gage, Kocour Co., Chicago, Ill.

[2] J. B. Mohler and R. A. Schaefer, "Laboratory Apparatus for Controlled Current Distribution on Small Flat Specimens," *Monthly Review*, Am. Electroplaters' Soc., Vol. 34, p. 1361 (1947).

DESIGN OF THE PROGRAM

To provide data permitting evaluation of the measurement errors associated with the Magne-Gage, Boonton gage, Dermitron, and Kocour gage, it was decided to have the three cooperating laboratories perform five determinations on each specimen taken from 15 test panels. The specimens were formed by sectioning the test panels. One specimen from each panel was sent to each laboratory and the fourth specimen kept in reserve for possible use at a later date. Identification of the specimens was made by letter code. The laboratories were given no information regarding the actual thicknesses of the specimens.

Laboratory 3 did considerably more work than requested by providing a total of 25 measurements on each specimen for each method except the Kocour gage. The 25 measurements consisted of five measurements made by each of five operators. This permitted an estimate of errors attributable to operators when using the same equipment. For all three laboratories, the Kocour gage measurements were made last since this method removes a small area of the plated surface.

Rather unfortunate experiences with test programs of this type indicated the necessity of certain safeguards being taken prior to undertaking the main experiment. The major safeguards were directed toward assurance that each test panel was plated uniformly throughout its entire surface. Each panel prior to sectioning was measured a number of times by a Magne-Gage at points randomly selected over its entire surface. None other than small chance variations could be detected. The manufacturer who supplied the thickest panel said that his tests indicated uniformity within $\pm 1.5 \times 10^{-5}$ in.

Another safeguard against variation between specimens from the same test panel was built into the experiment itself. This was achieved by having groups of three test panels made in so far as possible to the same nominal thickness. A laboratory which by chance might get a specimen cut from the thin portion of one test panel could hardly be unlucky enough to get the thin quarters of the other two panels. This fact should be remembered when interpreting the results.

One other preliminary precaution considered necessary was to send a trial specimen in the 0.5-mil thickness range to each cooperating laboratory several months prior to the experiment. This was done to guard against gross errors in method or equipment setup invalidating the main experiment. This may be criticized from a theoretical standpoint in that it could possibly make the three cooperating laboratories atypical of the major body of other laboratories, but is considered by those who designed the test as quite defensible on the grounds of practical necessity. The results of this preliminary test are of no importance except to note that no gross errors were found. No attempt was made to calibrate methods between laboratories with these trial specimens.

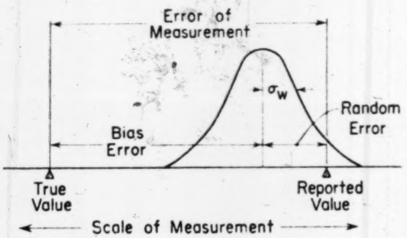


FIG. 1.—Diagram of Types of Error of Measurement.

TEST RESULTS

The data of this experiment have been analyzed in a manner which utilizes a viewpoint regarding errors of measurement borrowed from the field of statistics. To establish a clear understanding of the results presented, it becomes necessary first to consider briefly errors of measurement from this statistical point of view.

TABLE I.—RESULTS WITH MAGNE-GAGE (READINGS IN 10^{-5} IN.).

Test Panel Sample	Thickness by Weight Data	Laboratory 1			Laboratory 2			Laboratory 3			Difference Between Average and Weight Determination		
		Average of 5 Readings	High Value	Low Value	Average of 5 Readings	High Value	Low Value	Average of 25 Readings	High Value	Low Value	Laboratory 1	Laboratory 2	Laboratory 3
C	9.7	8.8	9	8	11.0	13	10	10.7	13	8	−0.9	1.3	1.0
H	9.7	8.2	9	8	11.0	13	10	10.2	13	6	−1.5	1.3	0.5
O	9.8	9.0	9	9	11.8	15	10	9.6	13	6	−0.8	2.0	−0.2
Average	9.7	8.7	...	...	11.3	...	...	10.2	...	...	−1.1	1.5	0.4
σ_w	...	0.29	...	...	1.58	...	...	0.74	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	0.80	...
A	24	24.4	27	23	24.2	25	24	25.1	28	21	0.4	0.2	1.1
F	23	23.6	26	22	24.2	28	22	22.5	26	17	0.6	1.2	−0.5
M	24	26.0	28	23	23.8	25	23	23.3	27	18	2.0	−0.2	−0.7
Average	24	24.7	...	...	24.1	...	...	23.6	...	...	1.0	0.4	0.0
σ_w	...	1.86	...	...	1.29	...	...	1.86	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.78	...
D	36	40.0	42	36	38.8	41	36	38.8	44	29	4.0	2.8	2.8
J	36	47.4	50	40	40.2	47	34	38.8	46	33	11.4	4.2	2.8
N	36	45.4	48	44	43.0	45	40	38.6	43	33	9.4	7.0	2.6
Average	36	44.3	...	...	40.7	...	...	38.7	...	...	8.3	4.7	2.7
σ_w	...	2.87	...	...	3.30	...	...	1.88	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	2.22	...
B	43	52.4	60	48	46.6	48	45	44.4	51	37	9.4	3.6	1.4
G	44	50.4	51	50	48.4	53	45	44.4	52	38	6.4	4.4	0.4
L	44	47.8	51	41	47.0	51	45	44.8	51	40	3.8	3.0	0.8
Average	44	50.2	...	...	47.3	...	...	44.5	...	...	6.5	3.7	0.9
σ_w	...	3.30	...	...	2.43	...	...	1.68	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	2.02	...
E	74	78.6	84	75	79.0	83	73	73.9	86	65	4.6	5.0	−0.1
K	74	78.4	79	77	75.6	79	72	76.2	86	67	4.4	1.6	2.2
P	74	78.2	82	75	81.4	87	73	74.4	89	64	4.2	7.4	0.4
Average	74	78.4	...	...	78.7	...	...	74.8	...	...	4.4	4.7	0.8
σ_w	...	2.58	...	...	4.44	...	...	2.63	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	2.88	...

Statistics recognizes that no device which measures in numerical units can be perfect. The value it reports differs from the true value of the characteristic measured by some small or large amount. This difference, or error of measurement, is for convenience broken into two types. The first is usually called random error or within-test error, while the second is commonly known as bias error. This idea is presented in Fig. 1.

TABLE II.—RESULTS WITH BOONTON GAGE (READINGS IN 10^{-5} IN.).

Test Panel Sample	Thickness by Weight Data	Laboratory 1			Laboratory 2			Laboratory 3			Difference Between Average and Weight Determination		
		Average of 5 Readings	High Value	Low Value	Average of 5 Readings	High Value	Low Value	Average of 25 Readings	High Value	Low Value	Laboratory 1	Laboratory 2	Laboratory 3
C	9.7	16.4	18	13	1.2	2	1	13.1	20	11	6.7	−8.5	3.4
H	9.7	13.5	16	12	10.4	12	10	8.0	11	4	3.8	0.7	−1.7
O	9.8	19.4	20	18	2.0	4	1	11.6	16	8	9.6	−7.8	1.8
Average	9.7	16.4	...	...	4.5	...	...	10.9	...	...	6.7	−5.2	1.2
σ_w	...	1.58	...	...	0.86	...	...	1.10	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.13	...
A	24	32.8	35	31	25.4	27	22	28.5	30	25	8.8	1.4	4.5
F	23	33.4	35	32	27.2	28	26	29.5	31	25	10.4	4.2	6.5
M	24	19.1	20	18	27.4	26	23	31.4	40	27	−4.9	0.4	7.4
Average	24	28.4	...	...	25.7	...	...	29.8	...	...	4.8	2.0	6.1
σ_w	...	1.29	...	...	1.43	...	...	1.37	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.37	...
D	36	43.3	45	41	39.4	40	39	39.1	41	36	7.3	2.3	3.1
J	36	44.2	45	43	38.2	40	35	40.2	42	38	8.2	2.2	4.2
N	36	44.0	45	43	39.4	40	39	39.2	41	35	8.0	3.4	3.2
Average	36	43.8	...	...	39.0	...	...	39.5	...	...	7.8	3.0	3.5
σ_w	...	1.15	...	...	1.01	...	...	1.11	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.10	...
B	43	50.6	55	49	50.2	52	48	46.0	49	40	7.6	7.2	3.0
G	44	49.1	50	49	50.0	51	49	46.9	51	42	5.1	6.0	2.9
L	44	50.4	52	50	49.8	51	48	46.7	51	42	6.4	5.8	2.7
Average	44	50.0	...	...	50.0	...	...	46.5	...	...	6.4	6.3	2.9
σ_w	...	1.29	...	...	1.29	...	...	1.52	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.45	...
E	74	78.6	80	76	99.4	100	99	81.2	86	72	4.6	25.4	7.2
K	74	78.8	80	76	94.8	100	89	81.3	89	72	4.8	20.8	7.3
P	74	79.6	82	76	98.8	99	98	80.5	86	74	5.6	24.8	6.5
Average	74	79.0	...	...	99.7	...	...	81.0	...	...	5.0	23.7	7.0
σ_w	...	2.01	...	...	1.86	...	...	1.63	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.72	...

Random error causes variability in results when repeated measurements are made on the same item under as nearly as possible identical testing conditions. The size of the variability due to this random error is measured by its standard deviation, identified as σ_w in Fig. 1. One can see that the averaging of a number of repeat measurements made under identical conditions would tend to reduce the random error but would leave unchanged the bias error associated with the chosen set of testing conditions. A testing device which has a small σ_w is considered to have good precision, although it may be quite inaccurate if bias error is present to any degree.

Bias error remains constant when repeated measurements are made over a short time period on the same item under identical testing conditions. It does have different values from laboratory to laboratory, instrument to instrument, operator to operator within a laboratory, and perhaps varies over long periods of time even with one operator using the same equipment in a laboratory. It is the type of thing one gets when an instrument is improperly calibrated or is operated under uniform but nonstandard conditions.

With the foregoing introduction, it is possible to interpret the results of the experiment as presented in Tables I through IV. The results of each test method are given on separate tables, as indicated by the table headings. For convenience, the values are recorded in 10^{-5} in. A value written as 24 would thus be 0.00024 in. Alternatively, a value of 24 could be looked upon as 0.24 mil.

TABLE III.—RESULTS WITH DERMITRON (READINGS IN 10^{-5} IN.).

Test Panel Sample	Thickness by Weight Data	Laboratory 1			Laboratory 2			Laboratory 3			Difference Between Average and Weight Determination		
		Average of 5 Readings	High Value	Low Value	Average of 5 Readings	High Value	Low Value	Average of 25 Readings	High Value	Low Value	Laboratory 1	Laboratory 2	Laboratory 3
C	9.7	4.4	5	4	5.8	6	5	7.8	9	7	−5.3	−3.9	−1.9
H	9.7	2.4	3	1	3.8	4	3	6.6	8	6	−7.3	−5.9	−3.1
O	9.8	4.4	5	3	5.6	6	5	6.6	8	6	−5.4	−4.2	−3.2
Average	9.7	3.7	...	...	5.1	...	...	7.0	...	...	−6.0	−4.7	−2.7
σ_w	...	0.72	...	...	0.43	...	...	0.20	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	0.31	...
A	24	18.0	19	17	19.0	19	19	19.9	21	19	−6.0	−5.0	−4.1
F	23	19.0	20	17	20.4	21	20	19.8	22	18	−4.0	−2.6	−3.2
M	24	19.2	20	18	19.6	21	19	20.3	22	18	−4.8	−4.4	−3.7
Average	24	18.7	...	...	19.7	...	...	20.0	...	...	−4.9	−4.0	−3.7
σ_w	...	1.00	...	...	0.43	...	...	0.60	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	0.63	...
D	36	31.4	35	28	32.0	32	32	31.8	34	29	−4.6	−4.0	−4.2
J	36	33.2	36	29	33.6	34	33	32.8	35	30	−2.8	−2.4	−3.2
N	36	32.8	34	32	33.6	34	33	33.7	36	30	−3.2	−2.4	−2.3
Average	36	32.5	...	...	33.1	...	...	32.8	...	...	−3.5	−2.9	−3.2
σ_w	...	2.29	...	...	0.28	...	...	0.43	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	0.67	...
B	43	39.6	41	38	40.0	41	39	40.9	43	37	−3.4	−3.0	−2.1
G	44	40.2	42	38	40.0	41	39	39.2	41	36	−3.8	−4.0	−4.8
L	44	40.2	42	38	39.6	40	39	40.3	42	38	−3.8	−4.4	−3.7
Average	44	40.0	...	...	39.9	...	...	40.1	...	...	−3.7	−3.8	−3.5
σ_w	...	1.58	...	...	0.72	...	...	0.63	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	0.78	...
E	74	70.2	74	68	71.0	71	71	71.0	74	68	−3.8	−3.0	−3.0
K	74	69.6	72	68	71.0	71	71	70.8	74	67	−4.4	−3.0	−3.2
P	74	70.0	72	68	71.0	71	71	70.2	72	66	−4.0	−3.0	−3.8
Average	74	69.9	...	...	71.0	...	...	70.7	...	...	−4.1	−3.0	−3.3
σ_w	...	2.01	...	...	0	...	...	0.57	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	0.70	...

The numbers in each row are the average of the readings obtained by a laboratory on the single specimen from each test panel. For each method but the Kocour gage (Table IV), laboratory 1 and laboratory 2 results are the average of 5 measurements, while laboratory 3 results are the average of 25 measurements. For the Kocour gage all three laboratory results presented are the average of 5 readings.

TABLE IV.—RESULTS WITH KOCOUR GAGE (READINGS IN 10^{-5} IN.).

Test Panel Sample	Thickness by Weight Data	Laboratory 1			Laboratory 2			Laboratory 3			Difference Between Average and Weight Determination		
		Average of 5 Readings	High Value	Low Value	Average of 5 Readings	High Value	Low Value	Average of 25 Readings	High Value	Low Value	Laboratory 1	Laboratory 2	Laboratory 3
C	9.7	10.2	11	10	10.0	10	10	9.2	10	8	0.5	0.3	−0.5
H	9.7	9.6	10	9	8.8	10	8	9.4	10	9	−0.1	−0.9	−0.3
O	9.8	9.6	10	9	9.6	10	9	9.2	10	8	−0.2	−0.2	−0.6
Average	9.7	9.8	...	...	9.5	...	...	9.3	...	...	0.1	−0.1	−0.5
σ_w	...	0.43	...	...	0.43	...	...	0.72	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	0.53	...
A	24	23.4	24	23	22.4	26	20	22.8	23	22	−0.6	−1.6	−1.2
F	23	24.0	25	23	23.4	26	22	22.4	24	21	1.0	0.4	−0.6
M	24	23.2	24	23	23.0	24	22	21.8	23	21	−0.8	−1.0	−2.2
Average	24	23.5	...	...	22.9	...	...	22.3	...	...	−0.1	0.7	−1.3
σ_w	...	0.57	...	...	1.72	...	...	0.86	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.05	...
D	36	34.8	35	34	34.8	36	33	33.4	34	33	−1.2	−1.2	−2.6
J	36	36.6	37	36	36.4	37	35	36.0	39	32	0.6	0.4	0
N	36	36.2	37	36	35.4	37	33	34.2	35	33	0.2	−0.6	−1.8
Average	36	35.9	...	...	35.5	...	...	34.5	...	...	−0.1	−0.5	−1.5
σ_w	...	0.43	...	...	1.29	...	...	1.43	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.05	...
B	43	43.2	46	42	42.4	45	39	42.2	44	40	0.2	−0.6	−0.8
G	44	44.2	45	43	39.8	41	38	41.4	43	40	0.2	−4.2	−2.6
L	44	42.2	43	41	41.8	43	39	42.2	43	41	−1.8	−2.2	−1.8
Average	44	43.2	...	...	41.3	...	...	41.9	...	...	−0.5	−2.3	−1.7
σ_w	...	1.15	...	...	1.86	...	...	1.29	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	1.43	...
E	74	72.6	74	71	75.8	79	73	71.6	73	69	−1.4	1.8	−2.4
K	74	72.0	73	71	70.8	73	68	68.0	72	65	−2.0	−3.2	−6.0
P	74	72.2	73	71	70.8	74	68	69.0	72	65	−1.8	−3.2	−5.0
Average	74	72.3	...	...	72.5	...	...	69.5	...	...	−1.7	−1.5	−4.5
σ_w	...	1.00	...	...	2.43	...	...	2.58	...	...	...	...	...
$\bar{\sigma}_w$	...	...	...	...	...	...	...	...	...	...	...	2.00	...

The test panels are arranged in groups of three according to thickness as determined by the weight change method. Similarities in thickness of test panels permit averaging results within each laboratory for these groups of three. This latter average of the averages, the grand average, represents 15 measurements for laboratories 1 and 2. For laboratory 3, it represents 15 measurements only on the Kocour gage, and 75 measurements in each other instance.

The differences between these several averages and the thicknesses determined by the weight change method for the same test panels are given at the right-hand side of each table. As an example, laboratory 1 averaged 8.8 on the specimen from test panel C when using the Magne-Gage, while the weight change method reported 9.7. The value −0.9 is the difference between these numbers, the negative sign indicating the Magne-Gage reported values lower than the weight change method.

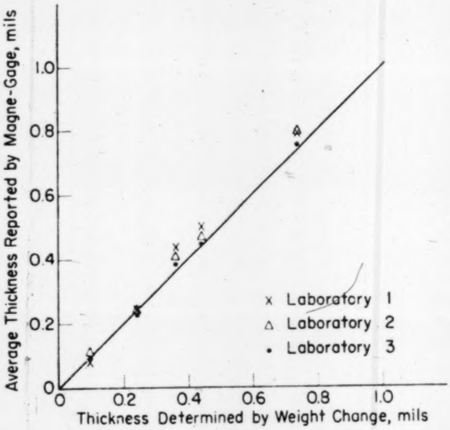


FIG. 2.—Average of Magne-Gage Determinations.

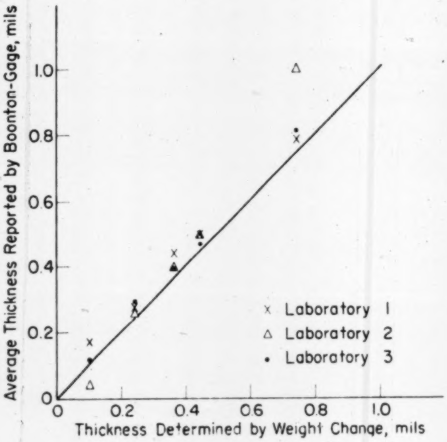


FIG. 3.—Average of Boonton Gage Determinations.

These differences between the average of each method and the weight change result may be considered estimates of the errors previously generically identified as bias errors. They must be treated as only estimates of the bias error because they necessarily contain the small portion of the random error which could not be removed by averaging. In most cases, the random error in this experiment was very small. The estimates of bias error are thus quite close to the true bias errors associated with the particular combination of laboratories, technicians,

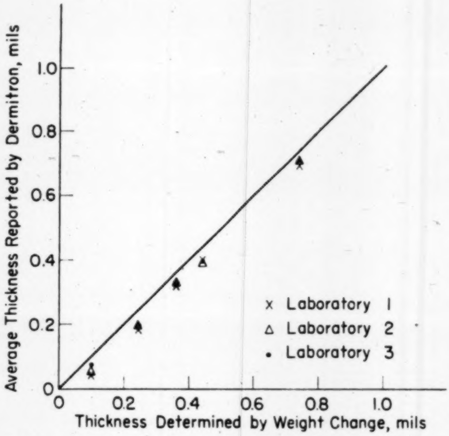


FIG. 4.—Average of Dermitron Determinations.

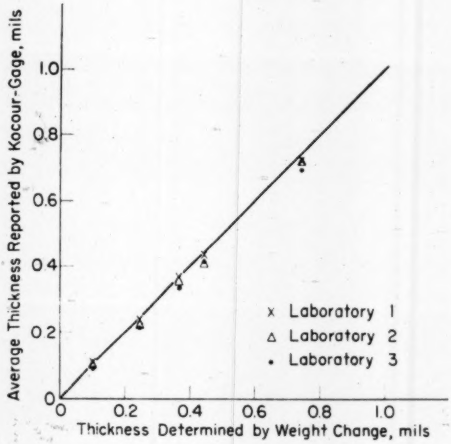


FIG. 5.—Average of Kocour Gage Measurements.

and testing instruments utilized in this particular experiment.

It should be mentioned that an attempt was made to estimate the magnitude of an operator's contribution to the total bias error. This attempt became involved with analysis of variance techniques and for clarity is best considered in another portion of the report.

Figures 2 to 5 give one a picture of the magnitude of bias error relative to the thickness as determined by weight change. The plotted points are the average readings for each laboratory, coded as indicated, plotted against the weight gain results. The diagonal line is not a curve of best fit through the points but shows where points would fall if one had a relationship free of bias error between the two methods. With perfection, we would, for example, have the Magne-Gage reading 0, 0.5, and 1.0 mil were the weight gain method to show 0, 0.5, and 1.0 mil on respective specimens. Estimated bias error on these figures is thus the difference measured vertically between each point and the diagonal line.

It is possible to establish the random error of the experiment quite independently of the bias error. One simple way of doing this is to determine the high and low values within each group of measurements made under identical conditions. The difference between high and low values is converted upon multiplication by a suitable factor into σ_w, the statistical estimate of random error. Tables I through IV present the high and low readings within each group. Because of the similarities involved, only one σ_w need be associated with each nominal thickness (comprising three test panels) at each laboratory. The grand average random error over all laboratories for each nominal thickness is identified as σ_w in the tables.

For these calculations it was necessary to recognize that laboratory 3 had 25 rather than 5 measurements. This was achieved by breaking each 25 measurements into five rational subgroups of 5 measurements each, and then using the high and low values within each of these subgroups. More sophisticated methods of estimating the random error when utilizing this body of data would, it is believed, present substantially the same results as indicated in the tables.

STATISTICAL ANALYSIS OF RESULTS

An analysis of variance study of the data is contained in this section. The report as a whole has been designed so that one may disregard this particular section without loss in continuity or meaning. The material should, however, be of interest to those familiar with the particular methodology involved.

It was considered inadvisable to group different testing devices and different nominal thicknesses into one analysis. Although this would have considerably reduced the analysis of variance calculations, it appeared that one could not, from physical considerations, justify the assumption that within-cell variances were identical. For this particular application it was thus necessary to perform two analyses of variance for each testing device at each nominal thickness. The first considered laboratories (3), samples (3) at each nominal thickness, and replications (5). The second considered operators (5), samples (3) at each nominal thickness, and replications (5).

We were concerned with a model II analysis of variance having two factors plus within-cell replication. This model could be stated in general mathematical terms in the following manner:

$$X_{ij\nu} = \xi + g_i + e_j + y_{ij} + z_{ij\nu}$$

where

ξ = a constant for all values of i, j, and ν,

g_i = the between row variation, distributed $N(0, \psi_1^2)$, with i going from 1 to k,

e_j = the between column variation, distributed $N(0, \psi_2^2)$, with j going from 1 to m,

TABLE V.—MODEL II.—TWO-WAY ANALYSIS OF VARIANCE WITH REPLICATION.

Factor	Degrees of Freedom	Mean Square	Components of Variance
Between rows $mn \sum_i^k (\bar{x}_{i..} - \bar{x}_{\cdots})^2$	$k - 1$	s_4^2	$\sigma^2 + n\omega^2 + mn\psi_1^2$
Between columns $kn \sum_j^m (\bar{x}_{.j.} - \bar{x}_{...})^2$	$m - 1$	s_3^2	$\sigma^2 + n\omega^2 + kn\psi_2^2$
Row + column $n \sum_i^k \sum_j^m (\bar{x}_{ij.} - \bar{x}_{i..} - \bar{x}_{.j.} + \bar{x}_{...})^2$	$(k - 1)\ (m - 1)$	s_2^2	$\sigma^2 + n\omega^2$
Replication $\sum_i^k \sum_j^m \sum_\nu^n (x_{ij\nu} - \bar{x}_{ij.})^2$	$km\ (m - 1)$	s_1^2	σ^2
Total $\sum_i^k \sum_j^m \sum_\nu^n (x_{ij\nu} - \bar{x}_{...})^2$	$kmn - 1$		

TABLE VI.—ANALYSIS OF VARIANCE TABLE.

For the Dermitron at 0.74 mil, original data in units of 10^{-5} in.

Source of Variance	Degrees of Freedom	Sample Size	Mean Square Values
Between laboratories..	2	13.911	6.955[a]
Between samples......	2	2.178	1.089
Sample × laboratory interaction.........	4	3.956	0.989
Replication (residual)..	36	47.200	1.311
Total...............	44	...	...

[a] Denotes significance on an F test at 0.05 mil.

y_{ij} = the row × column interaction, distributed $N(0, \omega^2)$, i and k as above, and

$z_{ij\nu}$ = the replication contribution, assumed distributed $N(0, \sigma^2)$, ν going from 1 to n, i, and j as above.

Using these symbols, the analysis of variance table became as shown in Table V.

If significance appeared on an F test between s_3^2 and s_2^2, or between s_4^2 and s_2^2, it was possible to partition the variances to obtain estimates of ψ_1^2 and ψ_2^2. The equations for this are:

$$E\,|\psi_1^2| = (s_4^2 - s_2^2)/mn,$$

$$E\,|\psi_2^2| = (s_3^2 - s_2^2)/kn$$

If $s_1^2 + s_2^2$ were not significantly different, they were pooled to obtain a better estimate of the within-cell replication variation. Although for brevity the complete calculations are not presented, the analysis of variance table involving laboratory and samples for the Dermitron data at 0.74 mil nominal thickness are given in Table VI as a sample.

Since between-laboratory variation was significant in this case, $E\,|\,t\,\psi_1^2\,|$ was also calculated:

$$\hat{s}_1^2 = \hat{s}_2^2 = \frac{47.200 + 3.956}{40} = \frac{51.156}{40} = 1.279$$

$$E\,|\,\psi_1^2| = \frac{6.955 - 1.279}{3 \times 5} = 0.378$$

$$\hat{\psi}_1 = (E\,|\,\psi_1^2\,|)^{1/2} = 0.378^{1/2} = 0.615$$

The mean square (MS) values for all of the analysis of variance tables have been summarized in Tables VIII and IX. Significant difference at the 5 per cent level from the within cell estimate of variance is indicated wherever it occurred. Tables X and XI tabulate the standard deviation, the square root of the variance components, in the cases where significance was indicated. These tables also contain the estimated standard deviations within cells. As before, the unit of measurement is 10^{-5} in.

TABLE VII.—RANDOM ERROR EXPRESSED AS A PERCENTAGE OF COATING THICKNESS.

Thickness, 10^{-5} in.	Magne-Gage		Boonton Gage		Dermitron		Kocour Gage	
	$\bar{\sigma}_w$	$\bar{\sigma}_w$ as per cent of Thickness	$\bar{\sigma}_w$	$\bar{\sigma}_w$ as per cent of Thickness	$\bar{\sigma}_w$	$\bar{\sigma}_w$ as per cent of Thickness	$\bar{\sigma}_w$	$\bar{\sigma}_w$ as per cent of Thickness
9.7	0.80	8.3	1.13	11.6	0.31	3.2	0.53	5.5
24	1.78	7.4	1.37	5.7	0.63	2.6	1.05	4.4
36	2.22	6.2	1.10	3.1	0.67	1.9	1.05	2.9
44	2.02	4.6	1.45	3.3	0.78	1.8	1.43	3.2
74	2.88	3.9	1.72	2.3	0.70	0.9	2.00	2.7

TABLE VIII.—TABULATION OF MEAN SQUARE VALUES: LABORATORY AND SAMPLE MAIN FACTORS.

Gage	Source of Variation	Nominal Thickness, 10^{-5} in.				
		9.7	24	36	44	74
Magne-Gage....	Between laboratories	29.405[a]	1.490	33.065	32.070[a]	135.470[a]
	Between samples	0.470	8.290	31.665	14.600	15.205
	Laboratories × samples	2.965[a]	7.022	22.335	9.165	13.867
	Replication	0.487	4.166	10.533	7.522	11.944
Boonton gage...	Between laboratories	522.155	52.155	109.155[a]	52.420	1546.070[a]
	Between samples	4.820	152.82	2.490	7.755	9.800
	Laboratories × samples	109.755[a]	92.957[a]	6.290[a]	21.690[a]	67.665[a]
	Replication	2.500	2.166	2.311	2.466	5.066
Dermitron......	Between laboratories	28.022[a]	3.622	1.800	1.866	6.955[a]
	Between samples	12.022[a]	2.022	8.865[a]	0.866	1.089
	Laboratories × samples	0.988[a]	1.688[a]	0.567	2.533	0.989
	Replication	0.322	0.588	1.766	1.100	1.311
Kocour gage....	Between laboratories	1.089	5.400[a]	7.222[a]	13.622[a]	40.289[a]
	Between samples	1.089	1.400	15.022[a]	2.489	41.689[a]
	Laboratories × samples	0.722	1.000	0.789	6.422	8.855
	Replication	0.444	1.644	1.500	2.444	4.600

[a] Significant difference at the 5 per cent level from the within-cell estimate of variance.

DISCUSSION OF RESULTS

The largest random error found had a standard deviation of 0.0288 mil (Table I) and was associated with the Magne-Gage method. This is small and is considered quite satisfactory for commercial measurement purposes. Figures showing random error as a percentage of sample thickness may be of some interest. They are presented in Table VII.

Bias error was in most cases either not present or was small enough to be neglected. In the few instances where bias error was somewhat serious, primarily with the Boonton gage used by laboratory 2, it appeared that calibration was probably at fault.

TABLE IX.—TABULATION OF MEAN SQUARE VALUES: OPERATOR AND SAMPLE MAIN FACTORS.

Gage	Source of Variation	Nominal Thickness, 10^{-5} in.				
		9.7	24	36	44	74
Magne-Gage....	Between operators	26.245	41.087[a]	93.800[a]	153.847[a]	633.580[a]
	Between samples	6.810	44.010[a]	3.235	1.480	37.485[a]
	Operators × samples	7.748[a]	2.246	7.175	9.446[a]	5.262
	Replication	0.680	3.920	4.326	2.980	7.433
Boonton gage...	Between operators	32.052[a]	28.422	5.620[a]	25.245	189.712[a]
	Between samples	171.375[a]	57.725	11.855[a]	4.970	3.045
	Operators × samples	1.873	30.767[a]	1.620	16.457[a]	43.321[a]
	Replication	2.206	2.240	1.653	2.733	4.040
Dermitron......	Between operators	11.246[a]	19.432[a]	23.845[a]	22.085[a]	53.653[a]
	Between samples	8.493[a]	2.080[a]	33.090[a]	18.120	3.880[a]
	Operators × samples	0.576[a]	0.914	1.928[a]	5.137[a]	0.714
	Replication	0.127	0.513	0.360	0.540	0.453

[a] Significant difference at the 5 per cent level from the within-cell estimate of variance.

TABLE X.—STANDARD DEVIATIONS ESTIMATED FROM VARIANCE COMPONENTS: LABORATORY AND SAMPLE MAIN FACTORS.

Gage	Source of Variation	Nominal Thickness, 10^{-5} in.				
		9.7	24	26	44	74
Magne-Gage.......	Between laboratories	1.327	...	...	1.275	2.867
	Between samples	...	...	...	...	...
	Laboratories × samples	0.703	...	...	...	...
	Replication	0.698	2.110	3.422	2.772	3.483
Boonton gage......	Between laboratories	...	...	2.619	...	9.928
	Between samples	...	...	...	...	...
	Laboratories × samples	4.632	4.261	0.892	1.961	3.538
	Replication	1.581	1.472	1.520	1.570	2.251
Dermitron.........	Between laboratories	1.342	...	...	...	0.615
	Between samples	0.857	...	0.694	...	...
	Laboratories × samples	0.365	0.469	...	...	...
	Replication	0.567	0.767	1.283	1.114	1.131
Kocour gage.......	Between laboratories	...	0.504	0.621	0.847	1.533
	Between samples	...	...	0.952	...	1.563
	Laboratories × samples	...	...	...	...	...
	Replication	0.687	1.257	1.195	1.686	2.242

Differences in technique from operator to operator seem capable of causing variations in results comparable to the small bias errors observed in the remaining instances. For substantiation of this contention, note in Table X that the Magne-Gage on 0.74 mil nominal thickness specimens reported a 0.02867 mil standard deviation between laboratories. Since different operators were used at each laboratory, this figure has, of course, incorporated within it differences between operators, as well as those between laboratories. Table XI indicates for the Magne-Gage at the same 0.74 mil thickness a standard deviation between operators of 0.06462 mil when using the same equipment. In other words, a separate experiment has indicated a variation due to operators *alone* more than twice that found between laboratories (and operators) in the first experiment. An examination of the data disclosed in this particular instance that one of the operators reported unusually high readings. Lack of experience with the Magne-Gage is considered one possible explanation for these aberrant readings.

TABLE XI.—STANDARD DEVIATIONS ESTIMATED FROM VARIANCE COMPONENTS: OPERATOR AND SAMPLE MAIN FACTORS.

Gage	Source of Variation	Nominal Thickness, 10^{-5} in.				
		9.7	24	36	44	74
Magne-Gage	Between operators	...	1.577	2.437	3.102	6.462
	Between sampies	...	1.269	...	...	1.101
	Operators × samples	1.189	...	...	1.137	...
	Replication	0.825	1.929	2.159	1.726	2.679
Boonton gage	Between operators	1.411	...	0.513	...	3.124
	Between samples	2.601	...	0.639	...	...
	Operators × samples	...	2.388	...	1.657	2.803
	Replication	1.472	1.497	1.284	1.653	2.010
Dermitron	Between operators	0.843	1.122	1.209	1.063	1.883
	Between samples	0.562	0.245	1.116	...	0.367
	Operators × samples	1.258	...	0.559	0.959	...
	Replication	0.356	0.748	0.600	0.735	0.696

TABLE XII.—ESTIMATION OF MAXIMUM SIZE OF DIFFERENCES BETWEEN AVERAGES.

(In units of 10^{-5} in.)

Gage	Number of Items Averaged			
	1	5	10	25
Magne-Gage	7.98	3.57	2.52	1.60
Boonton gage	4.77	2.13	1.51	0.95
Dermitron	2.16	0.97	0.68	0.43
Kocour gage	5.54	2.48	1.75	1.11

Another bit of useful information may be gleaned from the experiment herein reported. When laboratories compare results obtained upon measurement of the same or assumed identical items, the significance of observed differences in results is frequently of some consequence. The data of the present experiment permit estimation of the maximum size of differences between averages which could be due to random (chance) error alone (Table XII).

Differences found to be larger than those shown in Table XII would be a statistical indication of bias error which may, or may not, be considered of real consequence. One stands only a 1 in 20 chance (0.05) of being wrong when using the table in this manner.

SUMMARY

With proper calibration the Magne-Gage, Dermitron, Kocour gage and Boonton gage methods give equivalent results when measuring zinc on steel in the 0.1 to 1.0 mil range. Directions for the general use of magnetic gages are given in ASTM Method A 219[3] for thickness determination.

---

[3] Standard Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 – 58). 1958 Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE B-9

ON

METAL POWDERS AND METAL POWDER PRODUCTS*

Committee B-9 on Metal Powders and Metal Powder Products held one meeting during the year: in New York City on Feb. 16, 1960.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. L. Bonanno.

Vice-Chairman, H. R. Biehl.

Secretary, C. G. Johnson.

Advisory Committee Members-at-Large: A. S. Doty, F. Emley, F. V. Lenel, and G. L. Werley.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (**B 202 - 58 T**)[1] and continuation of the specifications as tentative:

Table I.—Change the range of copper in Type II, Class B from "5.0 to 30.0 per cent" to two ranges as follows: "7.0 to 11.0 per cent" and "18.0 to 22.0 per cent."

Table III.—Change the strength constant (K) for Type I, Classes A and B from the present "22,500" to "26,500."

Explanatory Notes.—Add new Notes 7 and 8 to read as follows:

NOTE 7.—*Flange and Thrust Washer Specifications*—Diameter and thickness specifications for flange and thrust washers are shown in Table X.

NOTE 8.—It was found that a circulating type oil containing rust and oxidation inhibitors is the most desirable type of oil to be used. The viscosity should be specified by the user in accordance with the application.

New Table.—Add a new Table X as shown in the accompanying Table I.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Bronze Sintered Metal Powder Structural Parts (**B 255 - 58 T**),[2] and

Brass Sintered Metal Powder Structural Parts (**B 282 - 58 T**).[2]

Tentative Method for:

Subsieve Analysis of Granular Metal Powders by Air Classification (**B 293 - 54 T**),[3] and

Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings (**B 328 - 58 T**).[3]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as appended hereto[4] of the Standard Method of Sampling Finished Lots of Metal Powders (**B 215 - 48**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 1.

[2] 1958 Book of ASTM Standards, Part 2.

[3] 1958 Book of ASTM Standards, Part 3.

[4] The revised standard appears in the 1960 Supplement to Book of ASTM Standards, Part 3.

TABLE I.—FLANGE AND THRUST WASHERS—DIAMETER AND THICKNESS SPECIFICATIONS.

(New Table X for Specifications B 202)

FLANGE BEARINGS: FLANGE DIAMETER TOLERANCES, IN INCHES

Diameter Range	Type I: Non-Ferrous Base		Type II: Ferrous Base	
	Class A	Class B	Class A	Class B
0 to 1½...	±0.0025	±0.005	±0.0025	±0.005
1½ to 3...	±0.005	±0.010	+0.005	+0.010
3 to 6.....	±0.010	±0.025	±0.010	±0.025

NOTE.—Normally the outside diameters of the flange are not too critical. Therefore, they should not be held too close—unless required. Class A tolerances may require additional operation and, therefore, are costlier.

FLANGE BEARINGS: FLANGE THICKNESS TOLERANCES, IN INCHES

Diameter Range	Non-Ferrous Base		Ferrous Base	
	Class A	Class B	Class A	Class B
0 to 1½...	±0.0025	±0.005	±0.0025	±0.005
1½ to 3...	±0.007	±0.010	±0.007	±0.010
3 to 6.....	±0.010	±0.015	±0.010	±0.015

NOTE.—See note above; the same applies.

THRUST WASHERS, ¼ IN. THICKNESS, MAX

O.D. tolerances same as for flange bearings. Thickness tolerances as follows for all diameters; in inches:

	Non-Ferrous Base		Ferrous Base	
	Class A	Class B	Class A	Class B
	±0.0025	±0.005	±0.0025	±0.005

PARALLELISM ON FACES, MAX

Diameter Range	Non-Ferrous Base		Ferrous Base	
	Class A	Class B	Class A	Class B
0 to 1½...	0.002	0.003	0.003	0.005
1½ to 3...	0.003	0.004	0.005	0.007
3 to 6.....	0.004	0.005	0.007	0.010

NOTE.—Closer tolerances can be held, but require secondary operations and therefore are costlier.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Green Strength of Compacted Metal Powder Specimens (**B 312 – 58 T**),

Apparent Density of Refractory Metals and Compounds by the Scott Volumeter (**B 329 – 58 T**),

Average Particle Size of Refractory Metals and Compounds by Fisher Subsieve Sizer (**B 330 – 58 T**), and

Compressibility of Metal Powders (**B 331 – 58 T**).

Tentative Recommended Practice for:

Hardness Testing of Cemented Carbides (**B 294 – 54 T**).

Tentative Specifications for:

Copper-Infiltrated Iron Sintered Metal Powder Structural Parts (**B 303 – 58 T**),

High Density Iron, Sintered Metal Powder Structural Parts (**B 309 – 58 T**), and

Low and Medium Density Iron, Sintered Metal Powder Structural Parts (**B 310 – 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ADVISORY COMMITTEE ACTIVITIES

The Advisory Committee held two meetings during the year. Committee personnel changes were reviewed and approved. O. W. Reen was appointed chairman of Subcommittee III-C on Cemented Carbides. H. R. Biehl was reappointed as chairman of the Award of Merit Committee. F. V. Lenel was appointed as official representative of Committee B-9 on the ASTM Coordinating Committee on Cermets. The activities of subcommittees were reviewed, and topics for extending the work of several committees were studied. The separate volume of the Committee B-9 specifications[6] is being well received.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

The seven active subcommittees and sections met twice during the year: in Chicago, Ill., on Nov. 2 and 3, 1959, and prior to the main committee meeting in New York City on Feb. 15 and 16, 1960. Subcommittee III-D on Friction Materials met an additional time, May 21, 1959, at Detroit, Mich.

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman) is planning to survey the committee on the proposal that the noun "sintering" be used as the general term for the end product of the powder metallurgy industry, and that definitions of other terms used to designate certain classes of sintered products be included. A task group headed by F. Emley is studying the standard tension test bar with the purpose of formulating proposals for design modifications of the bar. A task group was appointed, with P. V. Schneider as chairman, to study the area of impact testing for sintered products and to develop a recommendation. E. Geijer's committee reported the results of a study of the variation in density and tensile strength obtained by sintering identical test bars composed of iron (Ancor MH 100) + 0.5 per cent zinc stearate at twelve different laboratories. It is planned to prepare this report for publication in the Society's *Proceedings*. Future work of this task group is being directed toward a more quantitative study of the effect of the temperature variable.

Subcommittee II on Metal Powders (J. B. Haertlein, chairman):

Section A on Base Metal Powders (J. B. Haertlein, chairman) revised the Standard Method of Sampling Finished Lots of Metal Powders (B 215 – 48) as appended to this report.[4] A task force under P. V. Schneider has finished a study of round-robin tests on the effect of sintering conditions on the dimensional change of bronze powders. A paper is being prepared for the ASTM *Proceedings* giving the results of the work. The committee recommended the adoption as standard of Method B 293 and the continuation of Methods B 312 and B 331 as tentatives, as reported earlier in this report. A task group set up under Committee E-3 for Chemical Analysis has submitted to Committee E-3 a method for hydrogen loss. Methods for the analysis of metal powders and of acid insolubles are under consideration.

Section B on Refractory Metal Powders (W. H. Bleecker, chairman) is continuing its work on tungsten powder and is conducting a round-robin series on the turbidimetric test method using the photelometer and a revised procedure suggested by A. Michaels.

Subcommittee III on Metal Powder Products (*R. P. Koehring, chairman*):

Section A on Bearings (M. H. Meighan, chairman) prepared the specifications for flange and thrust washer diameters and the other revisions in Specification B 202 referred to earlier in this report. Work is continuing on the development of specifications for bronze on steel bearings, bushings, and thrust washers. The task force working on the correlation of K-factor, microstructure, and machinability of sintered bronze bearings is now preparing a paper on "The Microstructure of Sintered Bronze Bearings" for publication in the ASTM *Proceedings*.

Section B on Structural Parts (P. J. Shipe, chairman) is collecting information on iron-copper-carbon structural parts in order to issue a new specification or to reissue an existing specification to include this material. Task groups are working on a specification for high density iron-carbon material and studying methods for holding heat treated tension bars pursuant to obtaining mechanical data. The committee is recommending the adoption of Specifications B 255 and B 282 and Method B 328 as standard, and the continuation of Specifications B 303, B 309 and B 310 as tentative, as reported earlier in this report.

---

[6] 1959 Compilation of ASTM Standards on Metal Powders and Metal Powder Products (with Related Information).

A task group was appointed to study Specifications B 303, B 309 and B 310 to suggest uniform terminology in the name, to standardize terminology in the body of the standard, and to review the chemical analysis requirements. An editorial change is to be made in Specifications B 255 and B 282 consisting of deleting anything covered by the Test Method for Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil Impregnated Bearings (B 328 - 58 T).

Section C on Cemented Carbides (O. W. Reen, chairman) approved for letter ballot of the section the following Cemented Carbide Producers Assn. standards:

1. Tentative Recommended Practice for Determination of Transverse Rupture Strength of Cemented Carbides,
2. Tentative Recommended Practice for the Determination of Linear Thermal Expansion of Cemented Carbides,
3. Tentative Recommended Practice for the Determination of Electrical Resistivity of Cemented Tungsten Carbide,
4. Tentative Recommended Practice for the Determination of Compressive Strength of Cemented Carbides,
5. Tentative Recommended Practice for the Determination of Poisson's Ratio of Cemented Carbides, and
6. Tentative Recommended Practice for the Determination of Young's Modulus of Cemented Carbides.

The committee voted to retain Recommended Practice B 294 as tentative, as reported earlier in this report.

Section D on Friction Materials (H. B. Huntress, chairman) has made considerable progress in preparing specifications for density and transverse rupture of sintered metal friction materials. A recent letter ballot of the section indicated both of these specifications need rewriting and re-balloting. The section is working on both wet and dry friction testing, and decided at the last meeting to standardize on a test fixture for wet friction testing. Members of the committee from Raybestos-Manhattan accepted the responsibility for setting up procedures for cooperative dry friction testing and have submitted alternate test programs.

This report has been submitted to letter ballot of the committee, which consists of 78 members; 54 members returned their ballots, of whom 49 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JOSEPH L. BONANNO,
Chairman.

CARL G. JOHNSON,
Secretary.

REPORT OF ADVISORY COMMITTEE
ON
CORROSION

The Advisory Committee on Corrosion held two meetings during the year: on June 23, 1959, in Atlantic City, N. J., and on February 2, 1960, in Chicago, Ill.

PERSONNEL

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts has asked to be represented on the Advisory Committee by E. I. Shobert, II, and E. K. Camp. New committee representatives are as follows: for Committee B-3, W. H. Ailor replaces A. W. Tracy; for Committee B-7, R. A. Harris replaces I. V. Williams; for Committee C-19, R. F. Zemer replaces L. E. Johnson; and for Committee D-14, H. R. Butzlaff replaces D. A. George.

Mr. H. A. Pray has been recommended for reappointment as a member-at-large for a three-year term beginning June 1960.

STATUS OF ATMOSPHERIC EXPOSURE PROGRAMS

Committee A-5 on Corrosion of Iron and Steel:

Specimens are being prepared for a new exposure program to evaluate differences, if any, between the atmospheric corrosion resistance of individually dipped (from four conventional pots) as against continuously dipped (from six continuous galvanizing lines) zinc-coated roofing sheet. Aluminum-coated sheet specimens are also included. The 28 by 36-in. specimens will be exposed at State College, Pa.; Newark, N. J. (New York area); Kure Beach (800-ft site), N. C.; Point Reyes, Calif.; and Freeport, Tex., in September, 1960.

Specimens of aluminum-coated wire and fencing are being collected for a test that will supplement the 1936 test program that included zinc- and copper-coated steel, and stainless steel wire and fencing.

Committee A-10 on Iron-Chromium, Iron-Chromium- Nickel, and Related Alloys:

The joint NACE-ASTM report on information supplied by industry as to stress-corrosion cracking of austenitic stainless steels has been published as *ASTM Special Technical Publication No. 264.*

The 6-yr inspection data for chromium, chromium-manganese, and chromium-nickel stainless steel panels exposed at Pittsburgh, Pa., and Newark, N. J. (New York area) in 1953 have been collected. An intergranular corrosion test has been under investigation and preliminary data indicate that the nitric-hydrofluoric acid test may have considerable merit for the evaluation of type 316 L stainless steel.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

With the completion of the 1948 exposures for calibrating the relative corrosivity of 19 atmospheric test sites, a new calibration test program to cover 42 test sites covering those not included earlier has been initiated.

There will be 5 sites in England, 8 in Canada, and 4 in the Panama Canal Zone. A tie-in with a Philippine test site will also be initiated next year.

Continuous recording apparatus to measure temperature, time-of-wetness, and sulfur dioxide activity at corrosion sites will soon be initiated at 4 U. S. and 3 Canadian test sites. The data derived from this apparatus will be compared with data obtained from copper, steel, and zinc specimens exposed during the same period.

The 15-yr data from the 1941 exposures of galvanic couples involving 7 metals when coupled to two stainless steels[1] is now complete and a report is being prepared for publication. This test was initiated to study galvanic corrosion of aluminum and stainless steel when definitely coupled. There were some cases in the 1931 program[2] in which the aluminum and stainless steel did not form an electric contact.

A set of specimens exposed for 1 yr from the 1958 program of panel-type galvanic cell specimens using the bolted panel technique has been collected and is being tested for change in tensile strength.

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts:

A task group has been authorized to establish an atmospheric exposure program for static contacts and connections. The specimens will be mounted in louvered shelters and exposed in a rural, a marine, and an industrial atmosphere.

The committee is also maintaining liaison with Subcommittee 40-5 on Basic Testing Procedures of Technical Committee 40 on Components for Electronic Equipment, of the International Electrotechnical Commission. This group is working on corrosion testing of cadmium, nickel, and zinc on steel strip and plate specimens.

Committee B-6 on Die-Cast Metals and Alloys:

Aluminum die-casting alloy SC84A was prepared with five zinc contents: 0.25, 0.50, 1.0, 1.5, and 2.0 per cent. The 1500 bars thus prepared were placed on the Port of New York Authority Building, N. Y., and at the Kure Beach, N. C. (80-ft and 800-ft) sites for exposures of 1, 3, 6, and 12 years. The 6-yr exposure data for this test appear in the 1960 report of this committee.

The 20-yr specimens from the 1939 exposure of magnesium and zinc alloy bars have been collected to determine the change in weight, tensile strength, impact strength, dimensions, and hardness.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

The data gleaned from the 5-yr exposure period of sand and permanent mold castings of aluminum and magnesium alloys exposed in 1953 have been published.[3] Five-year data from the wrought specimens from the same program are also being prepared.

Committee B-8 on Electrodeposited Metallic Coatings:

A new Program No. 5, a development of the earlier atmospheric exposure

---

[1] "Report of Subcommittee VIII on Galvanic and Electrolytic Corrosion, Stainless Steels Coupled with Other Metals—5 Yr Exposure," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 167 (1948).

[2] Final report given in *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 247 (1939).

[3] Separate reprints are available.

TABLE I.—CORROSION TESTS OF IRON AND STEEL UNDERTAKEN BY ASTM COMMITTEES.

The boldface numbers refer to the references given at the end of this report.

	Architectural Sheet	Plate Specimens	Wire Specimens	Hardware	Pipe	Sandwich Panels	Galvanic Couples
Mild steel	A-5 (**1,3,8**)	A-5 (**2,5,7,9**) B-3 (**19**)	A-5 (**6**)	A-5 (**4**)	A-5 (**2,4**)	...	B-3 (**14,15,16, 18**)
Low-carbon steel	...	...	...	...	...	...	B-3 (**17**)
Copper-bearing steel	A-5 (**1,3**)	A-5 (**2,5**)	A-5 (**6**)	...	...	...	...
Silicon steel	...	A-5 (**2**)	...	...	...	...	...
Low-alloy steel	...	A-5 (**7**)	...	...	...	...	...
Stainless steel	A-10 (**11,13**)	A-10 (**10,12**)	A-5 (**6**) A-10 (**12**)	...	...	...	B-3 (**14,15,17, 18**)
Gray cast iron	...	...	...	A-5 (**4**)	...	...	...
Nodular iron	...	A-5 (**7**) A-7 (**9**)	...	...	...	...	...
Pearlitic iron	...	A-7 (**9**)	...	...	...	...	...
Malleable iron	...	A-5 (**7**) A-7 (**9**)	...	A-5 (**4**)	...	...	...
Wrought iron	A-5 (**1,3**)	A-5 (**5**) A-7 (**9**)	...	...	...	...	...
Aluminum coatings	A-5 (**8**)	A-5 (**7**)	A-5[a]	A-5 (**4**)	A-5 (**4**)	...	...
Cadmium coatings	...	...	...	A-5 (**4**)	A-5 (**4**)	...	B-3 (**17**)
Copper coatings	...	...	A-5 (**6**)	...	...	...	...
Lead coatings	...	...	A-5 (**6**)	A-5 (**4**)	A-5 (**4**)	...	B-3 (**17**)
Phosphate coatings	...	...	...	A-5 (**4**)	...	...	...
Porcelain enamel coatings	...	...	...	...	...	C-19 (**20**)	...
Zinc coatings	A-5 (**3,8**)	A-5 (**7**)	A-5 (**6**)	A-5 (**4**)	A-5 (**4**)	C-19 (**20**)	B-3 (**17**)

---

[a] New proposed program to be exposed by 1961.

programs, has been initiated. The program will develop further data for the study of the comparative performance of commercial and heavier chromium-nickel coatings on steel in order to determine which systems would lead to improved performance. The underplating will be multiple nickel plating with conventional chromium, crack-free chromium, and crack-free and conventional chromium overplating.

Specimens are being prepared for an atmospheric exposure to determine the performance of decorative chromium-nickel coatings on zinc die castings (Program No. 7).

Committee C-19 on Structural Sandwich Construction:

A portion of the data obtained from the 1955 exposures of sandwich construction panels sponsored by Committee C-19 is presented by R. F. Zemer, "Effect of Natural Environment on Sandwich Structures Using AMS 3722 Paper Honeycomb Core," Symposium on Durability and Weathering of Structural Sandwich Constructions, Am. Soc. Testing Mats., p. 9 (1960). (Issued as separate publication *ASTM STP No. 270.*)

Joint Committee on Leather:

A program to determine whether there is any correlation between bold atmospheric exposure of leathers in southern Florida and exposure in 5 types of laboratory apparatus has been completed. The data show that the time of year had a large influence on the degree of fading of samples exposed in southern Florida. Certain colored leathers exposed in summer suffered two to three times the fading as in winter exposures. The laboratory apparatus varied from no effect to less than the winter exposure in southern Florida.

CORROSION OF IRON AND STEEL

The work of the Society in the field of corrosion of iron and steel began with the motion of Edgar Marburg at the Annual Meeting of June, 1906: "In view of the importance of this subject and the lack of knowledge concerning the same, it would seem to be eminently proper for the Society to appoint a standing committee on the general subject of the corrosion of iron and steel. I move that the Executive Committee be requested to consider the desirability of appointing such a committee."

Committee U (now Committee A-5) was formed a few months later. In 1908 the committee began the first of an extended series of atmospheric exposure corrosion tests that have made significant contributions to knowledge of the corrosion of materials. Among the first studies was the famous laboratory sulfuric acid immersion test that proved that an acid-type test cannot be used as a reliable means of predicting atmospheric corrosion resistance.

Since that time corrosion studies of iron and steel have also been conducted by Committee A-7, exposing malleable irons, ductile irons, and steels; Committee A-10, exposing stainless steels; Committee B-3, exposing stainless and mild steels coupled to other metals to form galvanic cells; and Committee C-19, exposing stainless and porcelain enameled steels as facings of sandwich constructions.

A listing of the major corrosion studies utilizing iron and steel undertaken by ASTM technical committees is given in Table I and in the References following this report, and a brief description of each test program is given. Studies conducted by Committee A-5 on Corrosion of Iron and Steel are given in references (1) through (8). Committee A-7 on Malleable-Iron Castings has one atmospheric corrosion study currently exposed covering standard malleable irons, pearlitic, nodular, and heat-treated nodular irons, with mild steel control specimen (reference (9)). No data from this test are yet available. Those conducted by Committee A-10 on Iron-Chromium and Iron-Chromium-Nickel are given in references (10) through (13). Those conducted by Committee B-3 on Corrosion of Non-Ferrous Metals are listed in references (14) through (19). The results of the first atmospheric exposure study conducted by Committee C-19 are partially reported in reference (20).

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

S. F. ETRIS,
Secretary.

REFERENCES

(1) Copper additions (0.02 per cent max) 16- and 22-gage iron and steel roofing sheets. This study was made to determine the value of small copper additions to steel whose ore contained no trace copper, when such steels are exposed to corrosive atmospheres. Tests began in 1916, and were completed in 1954 at Annapolis, Md., Fort Sheridan, Ill., and Pittsburgh, Pa. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:
Analysis of metal: Vol. 16, I, p. 155 (1916), and Vol. 18, I, p. 160 (1918).
Final report: Vol. 54, p. 110 (1954).

Using extra duplicate samples from the above study, panels were immersed in running waters in Washington, D. C. city water, Severn River (at Annapolis, Md.), and Calumet Mine waters, Calumet, Pa. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:
Inspections begun: Vol. 20, I, p. 227 (1920).
Final report for Calumet Mine: Vol. 21, I, p. 157 (1921).
Final report for Washington, D. C.: Vol. 28, I, p. 160 (1928).
Final report for Severn River: Vol. 30, I, p. 223 (1930).
A critical study of the above tests: Vol. 29, II, p. 204 (1929).

The above copper- and non-copper-bearing sheets were immersed in running sea water at Portsmouth, N. H., and Key West, Fla. Results indicate that any beneficial effect of copper under totally immersed sea water conditions is so small as to be of no commercial importance. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:
First report: Vol. 27, I, p. 183 (1927).
Final statement: Vol. 50, p. 103 (1950).

(2) Riveted ship plate of copper and silicon steel with low- and high-copper steel, mild steel, and iron rivets, were immersed in sea water at Portsmouth, N. H., Key West, Fla., and Port Arthur, Tex., in 1926. Copper-bearing steel pipe (copper content from 0 to 1.0 per cent) were also immersed in sea water at Portsmouth, N. H., Key West, Fla., and Port Arthur, Tex., at the same time. The results were inconclusive and no final data were published. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:
First report on ship plate: Vol. 26, I, p. 129 (1926).
First report on steel pipe: Vol. 27, I, p. 183 (1927).
Final statement: Vol. 50, p. 103 (1950).

(3) Varying thicknesses of galvanizing on a variety of steels in the form of roofing sheets were placed on exposure in 1926 at Pittsburgh, Pa., Altoona, Pa., State College, Pa., Sandy Hook, N. J., and Key West, Fla. Wrought iron and bare steel sheets were also included. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:
Original data: Vol. 27, I, p. 191 (1927).
Final report for Pittsburgh, Pa.: Vol. 52, p. 111 (1952).
Final report for Key West, Fla., and Sandy Hook, N. J.: Vol. 54, p. 110 (1954).
Final report for Altoona, Pa.: Vol. 60, p. 113 (1960).
Latest report for State College, Pa.: Vol. 60, p. 114 (1960).

(4) Coated hardware of miscellaneous shapes

were exposed to the atmosphere in 1928 at Altoona, Pittsburgh, and State College, Pa.; Sandy Hook, N. J.; and Key West, Fla. Coatings were hot-dipped aluminum, lead, and zinc; electrodeposited zinc and cadmium; and phosphate coatings. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Materials exposed: Vol. 29, I, p. 149 (1929).
Final report for Altoona and Pittsburgh, Pa.: Vol. 44, p. 96 (1944).
Final report for Sandy Hook, N. J. and Key West, Fla.: Vol. 52, p. 119 (1952).
Latest report for State College, Pa.: Vol. 60, p. 116 (1960).

(5) The acid immersion test as an accelerated method of determining comparative atmospheric exposure corrosion was investigated in 1931. Twenty per cent sulfuric acid was used to test 10 types of iron and steel specimens cut from black sheets of Reference (**3**). Results showed that relative atmospheric corrosion cannot be predicated from the results of the acid test. Report published in *Proceedings*, Am. Soc. Testing Mats., Vol. 31, I, p. 176 (1931).

(6) Wire for farm-field fencing, barbed, chain-link, and strand was exposed in 1936 at 11 locations, fabricated from uncoated copper-bearing steel, zinc-coated copper-bearing steel, corrosion-resistant steel, copper-covered steel, and lead-coated steel. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Original data: Vol. 37, I, p. 122 (1937); Vol. 39, I, p. 156 (1939).
Final data for Sandy Hook and Brunot Island: Vol. 53, p. 102 (1953).
Data, 20-yr exposure: Vol. 57, p. 99 (1957).
Critical study of 20-yr data: Twenty-Year Atmospheric Corrosion Report on Metallic-Coated and Uncoated Wire and Wire Products (1960). (Issued as separate publication *ASTM STP No. 290.*)

(7) Hardware including carbon, 4 low-alloy steels, 2 nodular irons, and malleable iron coated with hot-dip zinc and aluminum, sprayed zinc and aluminum, and electroplated zinc. Atmospheric exposure initiated in 1958 at New York Area (Newark, N. J.) and 80- and 800-ft sites at Kure Beach, N. C. Report published in *Proceedings*, Am. Soc. Testing Mats., as follows:

Preliminary information: Vol. 59, p. 133 (1959).
First report: Vol. 60, p. 116 (1960).

(8) Corrugated steel roofing sheets (28 by 36 in.) coated with hot-dipped aluminum and zinc to be exposed in September, 1960, at 5 test sites. The test was designed to determine if there is a difference between the corrosion resistance of zinc applied by the continuous strip process and zinc applied by individually dipping each sheet. Six continuous galvanizing lines and four conventional galvanizing pots are represented.

(9) A series of standard malleable irons, pearlitic, nodular, and heat treated nodular irons (with mild steel control specimens) were exposed in 1958 at 5 sites to evaluate the effects of atmospheric corrosion on malleable iron specimens. Exposure periods are for 1, 3, 7, and 12 years. Date of initiation of tests given in *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 123 (1958).

(10) Development of corrosion tests for inspection of iron-chromium, iron-chromium-nickel, and related alloys. Salt-spray, boiling nitric acid, and the copper sulfate stain test were studied. Only the boiling nitric acid test is capable of furnishing accurate, reproducible, quantitative data. Report published in *Proceedings*, Am. Soc. Testing Mats., Vol. 33, I, p. 178 (1933).

(11) Stainless steel panels for architectural applications of chromium, chromium-manganese, and chromium-nickel compositions, exposed in 1953 in Pittsburgh, Pa., and in the New York area. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:

First year of exposure: Vol. 55, p. 173 (1955).
Third year of exposure: Vol. 56, p. 143 (1956).

(12) Stainless steel in 17 grades, including spot- and arc-welded panels, tension panels, Erickson cup panels, 0.505 in. tension test bar specimens, appearance panels, and fatigue coil springs both static and tension loaded. Exposed in 1958 at 6 exposure test sites. Preliminary report published in *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 160 (1959).

(13) Inspection of corrosion-resistant steels in architectural and structural applications. Some 26 buildings and the Reading Railroad "Crusader" have been inspected since 1937. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:

First report: Vol. 39, p. 197 (1939).
Latest report: Vol. 55, p. 160 (1955).

(14) Stainless steels, types 304 and 316, coupled with aluminum, copper, lead, zinc, bronze, monel, and mild steel to form galvanic cells. Exposed in 1941 at New York, N. Y.; Kure Beach, N. C.; and Altoona and State College, Pa., for 15 years. Reports pub-

lished in *Proceedings*, Am. Soc. Testing Mats., as follows:
Materials exposed: Vol. 42, p. 171 (1942).
Data, 5-yr exposure: Vol. 48, p. 167 (1948).
Final report in preparation.

(15) Disk specimens of carbon and type 304 stainless steel, and cadmium- and zinc-plated steel coupled to AZ31B magnesium alloys, were coupled to form galvanic cells and exposed at New York, N. Y., State College, Pa., Kure Beach, N. C., and the Canal Zone. Specimens were measured for change in weight. Complete report published in the Symposium on Atmospheric Corrosion of Non-Ferrous Metals, p. 89 (1955). (Issued as a separate technical publication *ASTM STP No. 175.*)

(16) Disk specimens of mild steel coupled with aluminum, copper, lead, nickel, tin, and zinc to form galvanic cells were exposed at 9 locations in 1931. The outer faces of the metal disks were protected by paint in order to confine the corrosion to the edges and the metal-to-metal surfaces of the couples. Reports published in *Proceedings*, Am. Soc. Testing Mats., as follows:
Data, 1-yr exposure: Vol. 34, I, p. 236 (1934).
Data, 3-yr exposure: Vol. 35, I, p. 167 (1935).
Data, 7-yr exposure: Vol. 39, p. 247 (1939).

(17) Threaded bolt and wire specimens of 15 metals and 6 metal platings were exposed in 4 atmospheric environments for 4 exposure periods in 1951. The results of this study appear in the Symposium on Atmospheric Corrosion of Non-Ferrous Metals, p. 116 (1955). (Issued as a separate technical publication *ASTM STP No. 175.*)

(18) Panels of dissimilar metals bolted together to form galvanic couples were exposed in 1958 at 4 test sites, in which 6 metals were coupled to 2 magnesium alloys. No data published to date.

(19) A study to determine the relative corrosivity of 20 atmospheric exposure test sites in terms of the corrosion rates of steel and zinc was begun in 1948 and completed in 1956. The final report appears in the *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 183 (1959). An additional study was begun in June, 1960, and covers 41 test sites in the United States, Canada, England, the Panama Canal Zone, and the Philippines.

(20) Structural sandwich constructions having porcelain enameled and galvanized steel faces with paper honeycomb cores; other facings of aluminum, fiber glass, glass, and kraft paper faces with paper and aluminum honeycomb cores; and bare paper honeycomb cores were exposed for 1-, 2-, and 3-yr periods beginning in 1955, at State College, Pa., and Kure Beach (80-ft site), N. C. Two similar tests were begun in 1957 and 1959.

Report on first test program: R. F. Zemer, "Effect of Natural Environment on Sandwich Structures Using AMS 3722 Paper Honeycomb Core," Symposium on Durability and Weathering of Structural Sandwich Constructions, Am. Soc. Testing Mats., p. 9 (1960). (Issued as a separate publication *ASTM STP No. 270.*)

REPORT OF THE JOINT ASTM-ASME COMMITTEE
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS*

Meetings:

Meetings of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals, its panels and subcommittees were held during the past year in connection with the annual meetings of ASTM in Atlantic City, N. J., June 26, 1959, and The American Society of Mechanical Engineers in Atlantic City Dec. 3, 1949. The Panel on Structural Materials for Airframes and Missiles and the Panel on Test Methods held a meeting at the ASTM Pacific Area National Meeting in October of 1959.

Technical Sessions and Papers:

A paper on the "Properties of 70-30 Copper-Nickel Alloy at Temperatures Ranging up to 1050 F," by W. F. Simmons, R. I. Jaffee, D. N. Williams, and B. J. Sirois[1] was sponsored by the Joint Committee and presented at the ASTM Annual Meeting in Atlantic City, June, 1959.

At the 1959 ASME Annual Meeting in December in Atlantic City, two sessions were cosponsored by the ASME Metals Engineering Division and the Joint Committee, at which seven papers were presented as follows:

"Some Observations on the Extrapolation of High-Temperature Ferritic-Steel Data," by Robert M. Goldhoff.

"The Creep-Rupture Properties of 80-Nickel 20-Chromium Alloys," by Robert Widmer and Nicholas J. Grant.

"The Extrapolation of Families of Curves by Recurrence Relations, with Application to Creep-Rupture Data," by Alexander Mendelson and S. S. Manson.

"Fabrication and Annealing Factors Affecting Grain Size of 18 Cr 8 Ni-Ti Superheater Materials in Steam Boilers," by Fritz Eberle and James S. Makris.

"Plasma Flame-Spraying Equipment Development," by Robert M. Nadler.

"The Role of Atmosphere on the Creep-Rupture Behavior of 80-Nickel 20-Chromium Alloys," by Robert Widmer and Nicholas J. Grant.

"Survey of Various Special Tests to Determine Elastic, Plastic, and Rupture Properties at Elevated Temperatures," by Frank Garofalo.

Membership:

Resignations from Mr. D. B. Rossheim, of the M. W. Kellogg Co., and Mr. J. A. Cerow, of the Chapman Valve Manufacturing Co., were received during the year.

Mr. T. E. Kihlgren, of the International Nickel Co., Inc., was elected to membership on the committee.

At the June meeting, a wrist watch was presented as a token of appreciation to Mr. V. T. Malcolm, retired Chairman of the Joint Committee.

Finances:

The status of funds in the committee's account as of Jan. 31, 1960, is given in Table I.

ACTIVITIES OF PANELS AND PROJECT SUBCOMMITTEES

Finance Subcommittee (N. L. Mochel, chairman):

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Published in *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 1035 (1959).

A campaign to raise $150,000 was initiated in June (authorized at the Dec., 1958 meeting). This fund was sought to cover the current needs of the committee and to support activities projected over the next five years, as fully outlined in a brochure accompanying the letters of solicitation. At the December meeting of the committee Mr. Mochel reported that the results of this campaign to Dec. 3, 1959, have been most satisfactory.

TABLE I.—STATUS OF FUNDS IN THE ACCOUNT OF THE ASTM-ASME JOINT COMMITTEE ON EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS AS OF JANUARY 31, 1960.

Balance on hand, January 31, 1959	$50,858.86
Receipts, through January 31, 1960	78,568.88
Total	$129,427.74
Disbursements through January 31, 1960	43,315.52
Balance on hand, January 31, 1960	$86,112.22

Disbursement by Projects:	*Contract Term*	*Contract Price*	*Paid*	*Balance*
Steam Power Panel:				
SP-5, Use of Austenitic Alloy Steels in Steam Line Service (Univ. of Michigan)	April 26, 1958 to April 25, 1960	$20,000.00	$16,427.73	$3,572.27
SP-6, Use of Austenitic Stainless Steels in Superheater Tubes (Univ. of Michigan)	Feb. 1, 1959 to Jan. 31, 1960	20,000.00	15,038.63	4,961.37
Data and Publication Panel:				
DP-9, Aluminum and Magnesium Alloys (Univ. of Michigan)	March 15, 1958 to Aug. 31, 1960	5,000.00	5,219.98	...
DP-10, Physical Properties of Metals (Battelle Memorial Institute)	...	...	2,250.00	...
DP-11, -13, Punch Card Tabulation of Data (Battelle Memorial Institute)	Dec. 19, 1956 to April 30, 1959	3,000.00	2,946.26	54.74
Test Methods Panel:				
TM-1, Calibration Specimens	...	...	1,500.00	...
				8,588.38
				$77,523.84
Allocated for further contract:				
SP-5, University of Michigan		$7,500.00	...	
SP-6, University of Michigan		20,000.00	...	
DP-9, University of Michigan		600.00	219.98	
DP-11 and 13, Battelle Memorial Inst.		1,000.00	...	
Total				28,880.02
Estimated uncommitted balance, January 31, 1960				$48,643.82

Panel on Structural Materials for Airframes and Missiles (J. J. Heger, chairman):

Meetings of this panel were held in June at Atlantic City and in October at San Francisco. The program and activities of the panel are organized as follows:

Task Group AM-2, Effect of Temperature on Elastic Constants.—In order to fulfill a need for more data on elastic modulus at elevated temperature, this group is cooperating with the Test Methods Panel on the standardization of methods for determining elastic constants.

Other task groups include: Task Group AM-3, Elevated Temperature Compression Properties of Sheet Materials; Task Group AM-4, Properties of Refractory Metals; and Task Group AM-5, Low-Temperature Properties of High Strength Aircraft and Missile Materials.

Data and Publications Panel (George V. Smith, chairman):

This panel met on June 24 and again on Dec. 3, and the status of its activities may be summarized as follows:

Project DP-9, Survey of Aluminum and Magnesium Alloys.—This survey is completed and a manuscript of the report has been prepared for publication.

Project DP-10, Effect of Temperature on Selected Physical Properties of Metals.—A contract to the sum of $7500 with Battelle Memorial Institute was made and work commenced on Nov. 1 on the evaluation of available data as covered in a previous report.[2] The anticipated completion date of the contract is seven months from Nov. 1.

Project DP-13, Continuing Data Survey and Punch Card Tabulation of Data.—A plan for the classification of high-temperature data for metals, presented on punch cards, has been developed by the panel. The committee has approved an additonal appropriation of $1000 for continuation of Projects DP-11 and DP-13 at Battelle Memorial Institute. Under the plan, data will be classified into three categories, and it is expected that ASTM will undertake the distribution of groups of data cards at intervals depending upon the rate of accumulation of data cards.

Gas Turbine Panel (Bruce O. Buckland, chairman):

Meetings of the Gas Turbine Panel were held on June 23 and Dec. 2. As directed by the Technical Advisory Committee on March 17, 1959, a study was made to appraise gas turbine problems as they relate to the activities of the Joint Committee. As a result of these studies, based upon the returns from a questionnaire sent out in May, it was recommended that the Gas Turbine Panel be continued and that efforts be made to obtain more "user" participation.

A task group was set up to explain the activities of the panel to users of gas turbines in the commercial aviation field.

A further task group was set up to examine some 50 suggested problems for study and make a selection of a relatively few (possibly 2 to 4) items, clearly important to the industry and gas turbine users.

Applied Research Panel (A. W. Rankin, chairman):

Meetings of the panel were held on June 23 and Dec. 1.

The panel has outlined a project on the Role of Surface and Material Defects in Thermal Fatigue and is attempting to secure an estimate of the cost involved.

The panel is studying a project for obtaining very long-time (over 30,000 hr duration) creep and rupture data. A survey is being undertaken to learn which organizations have such data, have such tests in progress, or would be willing to commit furnaces to a cooperative program.

The panel plans to hold an open panel meeting at the 1960 Annual ASTM Meeting devoted to discussions of low-cycle fatigue.

Low Temperature Panel (A. L. Tarr, chairman):

Meetings of the panel were held on June 25 and Dec. 2.

---

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 339 (1959).

Project LT-2, Literature Survey on Mechanical Properties of Useful Engineering Metals and Alloys at Low Temperatures planned by the panel was placed on inactive status in view of the National Bureau of Standards' scheduled publication "Mechanical Properties of Structural Material at Low Temperatures."

The panel has developed improved liaison coordination with other groups working on activities related to the mission of the Low Temperature Panel. Clarification is needed to determine the relation of the field of activities of the Low-Temperature Panel to that of ASME Research Committee on the Prevention of Fracture in Metals.

The committee has been alerted to the efforts of ASTM Committee A-1 on Steel, Subcommittee VI on Forgings, to establish transition temperature test methods for forgings. Subcommittee VI has in draft a method using a 50 per cent fibrous fracture appearance of a Charpy V-notch bar as a criterion.

The panel intends to sponsor a symposium on "Evaluation of Metallic Materials in Design for Low Temperature Service," to be held during the ASTM Annual Meeting in June of 1961.

Petroleum and Chemical Panel (C. L. Clark, chairman):

The panel met on June 23 and is active on a project on High-Temperature Strength of Welded Joints. Tests on this project have been completed by cooperating laboratories and it is anticipated that the panel will submit a final report.

Steam Power Panel (Paul Brister, chairman):

The panel held meetings on June 24 and Dec. 2. Progress of its projects is as follows:

Project SP-5, Use of Austenitic Alloy Steels in Steam Line Service.—Work to date under contract at the University of Michigan has been concentrated on investigating the differences between specimens which are known by experience to exhibit characteristics regarding cracking at or near welds, some being good and some being crack sensitive. The following significant information has resulted:

1. The R.P.I. testing method for discriminating between good and bad materials has been demonstrated as satisfactory, providing recognition is given to the sensitivity of results due to slight variations in testing procedures.

2. On specimens which show low ductility, an unidentified "eutectic" structure has been observed. It has not been observed on specimens with good ductility under the same testing conditions. Whether all materials that exhibit bad characteristics show this structure is not now known. Further investigation of this structure will be made.

3. Effects of prior history are small compared to heat-to-heat compositional differences. High-purity alloy laboratory heats showed much more resistance to the eutectic formation than commercial heats.

At the December meeting, an additional appropriation of $7500 was approved to complete the project.

Project SP-6, Use of Austenitic Stainless Steels in Superheater Tubes.—Work under contract at the University of Michigan has been concentrated on establishing the basic factors of physical metallurgy governing the strength of type 321 18-8 Ti stainless steel at elevated temperatures. Structural studies are in progress on reduced and cold drawn type 321 tubing supplied in the cold-worked condition. Specimens were annealed at temperatures ranging from 1650 to 2300 F, and the structures as related to creep properties are being established by optical and electron microscopy and by X-ray and electron diffraction techniques. Concurrently, laboratory heats were made with controlled compositional variations so that the minor as well as the major elements can be studied. Vacuum melting with carbon deoxidation has been used to control the effects of so-called deoxidants. An additional $20,000 was appropriated at the December meeting for the continuation of the research program at the University of Michigan.

The objective of the SP-6 research is to determine the basic causes for variation at high temperature in the properties of austenitic steel in the form of superheater tubing as used in the steam power industry. Classification of the basic metallurgy of such alloys is expected to provide a basis for avoiding unexpected failures during service by allowing the unit efficient use of existing alloys, as well as to provide information useful for the development of improved alloys.

(Projects SP-5 and SP-6 have been carried out in a large measure through the support of the Edison Electric Institute.)

Project SP-8, Effect of Long-Time Service on Elevated Temperature Properties of Tubes and Pipes.—A program is being developed with Edison Electric Institute to obtain materials from the utilities with significant service history and which may be evaluated for long time effects.

Test Methods Panel (J. W. Freeman, chairman):

The panel held meetings June 22, Oct. 13 and Dec. 2, and has been active on the following items:

1. The Recommended Practices for Conducting Creep and Creep Rupture Tension Tests of Metallic Materials Under Conditions of Rapid Heating and Short Time (E 150 – 59 T) and for Tension Tests of Metallic Materials at Elevated Temperatures with Rapid Heating and Conventional or Rapid Strain Rates (E 151 – 59 T) have been published by ASTM.[3] Some suggestions for improvement of these recommended practices are being reviewed for appropriate action at the ASTM Annual Meeting. These methods were developed through a request from the Materials Advisory Board of the National Academy of Sciences and in cooperation with the Aerospace Industries Association's aircraft and testing committees.

2. *Project TM-2, Calibration Specimens for Creep-Rupture Testing.*—Under a $1500 contract, the National Bureau of Standards has carried out a program of calibration upon a heat of type 304 steel (18 per cent chromium, 8 per cent nickel) selected to be distributed through ASTM Headquarters for calibration purposes, on a cost fee basis. The NBS has developed a statistical basis for evaluating the variations to be expected from specimens selected from various locations relative to the original ingot. A procedure has been developed to further verify the material for the Test Specimen Bank, and four cooperating laboratories are checking adjacent specimens from various ingot locations. The panel is studying a plan to accumulate the results of calibrations when the material from the bank becomes available for general distribution this year.

3. A task group is charged with developing a recommended practice for conducting high-speed, high-heating rate compression tests, as requested by the Materials Advisory Board, for action at the June, 1960, ASTM meeting.

4. A task group is charged with determining the feasibility and need for a recommended practice for compression and shear creep tests and, if needed, to prepare such a practice.

---

[3] 1959 Supplement to Book of ASTM Standards, Part 3.

5. A task group is planned for the preparation of a recommended practice for determining elastic modulus values (both dynamic and static) as requested by the Panel on Structural Materials for Airframes and Missiles.

6. A paper is being prepared on the methods used by the NBS to calibrate creep-rupture units.

7. The panel has under study a recommended practice for notched specimen rupture tests.

8. The panel has under study improved pyrometry practices for elevated temperature testings.

International Symposium on Creep and Rupture (N. L. Mochel, secretary):

Under the joint sponsorship of the ASME Metals Engineering Division and ASTM, an International Symposium on Creep and Rupture is scheduled to be held at the Hotel Biltmore, New York City, in August, 1963. Offers of papers and requests for information regarding this event should be directed to ASTM Headquarters, attention of N. L. Mochel, Secretary of the Symposium.

ASME Guidance Committee on the Mechanical Behavior of Metals:

This committee, organized under the chairmanship of Mr. Carl Schabtach, is charged with the review of ASME research proposals in the field of metals behavior, with the objective of minimizing duplication of effort. Although the activities of the Joint ASTM-ASME committee will remain autonomous, several of the committee's members will maintain a liaison relation with the ASME group.

This report has been submitted to letter ballot of the committee, which consists of 32 members; 22 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. KANTER,
Chairman.

HOWARD C. CROSS,
Secretary.

REPORT OF COMMITTEE C-1
ON
CEMENT*

Committee C-1 on Cement held two meetings during the year: in Atlantic City, N. J., on June 26, 1959, and in San Francisco, Calif., on Oct. 13, 1959.

The committee records with sorrow the deaths of three of its members: D. G. Miller, an Honorary Member, who joined in 1938; F. P. Diener, representative of the Universal Atlas Cement Division, United States Steel Corp., who joined in 1944; and P. D. Miesenhelder, representative of the Association of American Railroads, who first joined in 1933.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. R. Litehiser.
Vice-Chairman, W. S. Weaver.
Secretary, W. J. McCoy.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1959 Annual Meeting, Committee C-1 presented to the Society, through the Administrative Committee on Standards, the recommendation that the Tentative Method of Test for Compressive Strength of Hydraulic Cement Mortar (Using Portions of Prisms Broken in Flexure) (**C 349 - 57** T) be revised.

This recommendation was accepted by the Standards Committee on October 27, 1959, and the revised tentative method appears in the 1959 Supplement to the Book of ASTM Standards, Part 4.

New Tentatives

The committee recommends for publication as tentative the following methods of test as appended hereto:[1]

Tentative Methods of Test for:

False Set of Portland Cement (Paste Method), and

Potential Sulfate Resistance of Portland Cement.

Revision of Tentatives

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Sampling Hydraulic Cement (C 183 - 58 T):[2]

Section 3.—Change Paragraphs (*b*) and (*f*) to read as follows:

(*b*) *From Bulk Storage at Points of Discharge.*—Cement shall be withdrawn from the discharge openings in a steady stream until markers placed on top of the cement above each discharge opening pass through the opening. If a high circular silo is being sampled, all samples may be taken from one opening. If the number of barrels of cement in the bin exceeds 6000 when low rectangular bins are being sampled, the number of discharge openings employed in sampling shall be such that for no opening shall the number of samples represent more than one-half the contents of the bin or more than 10,000 barrels. While the cement is flowing through the openings, samples shall be taken at such intervals that one 5-lb test sample shall be secured for each 500 barrels in the bin or silo.

(*f*) *Protection of Samples.*—As samples are taken, they shall be placed directly in moisture-proof, airtight containers to avoid moisture absorption and aeration of the sample. If the samples are placed in cans, the cans shall be completely filled and sealed immediately. Moisture-proof multiple-wall paper bags or plastic bags may be used if they are strong enough to avoid breakage, and if they can be sealed immediately after filling in such a manner as to eliminate excess air in the sample, and avoid moisture absorption and aeration of the sample. The containers shall be numbered consecutively in the order in which the samples are taken. Samples taken by either the grab or composite method shall be treated as described herein.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

Section 4 (*a*).—Change to read as follows:

(*a*) Samples shall be packed by the purchaser in moisture-proof, airtight containers, which shall be crated, if necessary, by the manufacturer and shipped at the expense of the purchaser. Before testing, samples shall be passed through a No. 20 (840-micron) sieve in order to mix the sample, break up lumps, and remove foreign materials. Foreign materials and hardened lumps that do not break up on sieving or brushing shall be discarded. The cement shall be stored in airtight, moisture-proof containers to prevent aeration or absorption of moisture prior to test.

Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 – 58 T):[2]

Section 2.—Change the title of Paragraph (*a*) to read "*Portland Blast-Furnace Slag Cement*" and delete the second sentence in the present paragraph.

Change the title of Paragraph (*b*) to read "*Air-Entraining Portland Blast-Furnace Slag Cement.*"

Add a new Paragraph (*d*) to read as follows:

(*d*) *Portland Cement Clinker.*—The manufacturer may use any type of portland cement clinker suitable for use in manufacture of cements complying with the Specifications for Portland Cement (ASTM Designation: C 150), which in combination with the granulated blast-furnace slag will produce a product meeting the chemical and physical requirements specified in Sections 5 and 6, except that when moderate sulfate resistance is specified the tricalcium aluminate ($3\,CaO \cdot Al_2O_3$) content of the portland cement clinker shall not exceed 8 per cent.

Section 4 (*c*).—Replace the term "(type IS-A)" with the term "(type IS-A or type IS-A (MS)."

Section 5.—Retain Paragraph (*a*) as Section 5 and delete Paragraph (*b*).

Note 3.—Revise to read as follows:

NOTE 3.—The following are typical ranges in chemical composition of granulated blast-furnace slag:

Silicon dioxide (SiO_2), per cent.....	30 to 40
Aluminum oxide (Al_2O_3), per cent...	8 to 18
Ferrous oxide (FeO), per cent......	0 to 1
Calcium oxide (CaO), per cent.....	40 to 50
Magnesium oxide (MgO), per cent..	0 to 12
Manganic oxide (Mn_2O_3), per cent..	0 to 2
Sulfide sulfur (S), per cent.........	0 to 2

Table I.—Delete the phrase "Magnesium oxide (MgO), max, per cent ....5.0."

Table II.—Change the column headings and the values for air permeability and heat of hydration requirements to read as shown in the accompanying Table I; delete the present tensile strength requirements; and replace the present Footnote *c* with the new footnote shown in Table I.

TABLE I.—REVISIONS OF TABLE II, SPECIFICATIONS C 205.

	Type IS	Type IS-A	Type IS (MS)	Type IS-A (MS)
Specific surface by air permeability apparatus, sq cm per g:				
Average value, min...	2800	2800	2800	2800
Minimum value, any one sample..............	2600	2600	2600	2600
Heat of hydration:[c]				
7 days, maximum cal per g............	70	70	70	70
28 days, maximum cal per g............	80	80	80	80

[c] Applicable only when moderate heat of hydration (MH) is specified, in which case the strength requirements shall be 80 per cent of the values shown in the table.

Section 12(*j*).—Delete Paragraph (*j*), relettering the present Paragraph (*k*) as (*j*).

Tentative Method of Test for Fineness of Hydraulic Cement by the No. 325 Sieve (C 430 – 59 T):[3]

Section 2 (a).—In line 3, change "2 in." to read "2 ± 0.25 in." In line 4, change "3 in." to "3 ± 0.25 in." In line 12, change "18" to read "12" per cent.

Section 3.—In line 3, insert the words "clean, dry" before the term "No. 325 sieve."

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Specifications for Masonry Cement (C 91 – 59):[2]

In order to improve the procedures for testing masonry cement, the following revisions are recommended:

Section 4.—Change to read as follows:

4. *Staining.*—This requirement shall apply only when the invitation for bids specifically states that the cement shall be non-staining to limestone. Non-staining cement shall contain not more than 0.03 per cent of water-soluble "alkali" when determined in accordance with Section 30 of these specifications (Note 1).

Section 14.—Change to read as follows:

14. Soundness shall be determined in accordance with the Method of Test for Autoclave Expansion of Portland Cement (ASTM Designation: C 151), except that the paste shall be mixed according to Section 11 of these specifications and, after molding, the bars shall be stored in the moist cabinet for 48 hr ± 30 min before removal from the molds for measurement and test in the autoclave. The difference in length of the test specimen before and after autoclaving shall be calculated to the nearest 0.01 per cent of the effective gage length and shall be reported as the autoclave expansion of the masonry cement.

Section *20.*—Change Paragraphs (*a*) and (*b*) to read as follows:

20. (*a*) *Proportions for Mortar.*—Mortars for compressive strength, water retention, and air entrainment shall be proportioned to contain the weight of cement, in grams, equal to six times the printed bag weight in pounds and 1440 g of sand. The sand shall consist of 720 g of graded Ottawa sand and 720 g of 20–30 Ottawa sand (Note 2). The quantity of water, measured in milliliters, shall be such as to produce a flow of 110 ± 5 as determined by the flow table.

(*b*) *Mixing of Mortars.*—The mortar shall be mixed in accordance with Section 6 of Method C 305. If the mortar has the correct flow, another portion of the mortar shall be used for the determination of entrained air. After determination of the flow and the entrained air, the mortar shall be returned to the bowl and remixed 15 sec at the medium speed. Cubes shall then be made in the manner prescribed in Section 21.

Note 2.—Delete the present Note 2, renumbering subsequent Notes accordingly.

Section 28 (*b*).—In the ninth sentence, replace the word "trowel" with the word "straightedge," and change the tenth sentence to read: "Then cut off the mortar to a plane surface flush with the rim of the dish by drawing the straightedge with a sawing motion across the top of the dish in two cutting strokes, starting each cut from near the center of the dish."

Section 30.—Change to read as follows:

30. (*a*) To 150 g of sample in a 400-ml beaker, add 250 ml of distilled water. Stir thoroughly, let stand 30 min at room temperature, and stir again. Filter through a Buchner funnel, transfer the insoluble matter to the original beaker and rinse the funnel with 150 ml distilled water into the beaker containing the insoluble matter. Stir thoroughly, let stand 30 min at room temperature, and stir again. Filter as above and again return the insoluble matter to the original beaker. Wash the funnel with 100 ml distilled water into the beaker containing the insoluble matter. Stir thoroughly, let stand 30 min at room temperature, and stir again. Filter as above. Acidify the filtrate with 5 ml of concentrated

---

[3] 1959 Supplement to Book of ASTM Standards, Part 4.

HCl. Stir until cloudiness disappears. Evaporate the solution to about 400 ml. Cool to room temperature and transfer quantitatively to a 500-ml volumetric flask. Dilute to 500 ml. Transfer a 50-ml aliquot of this solution to a 100-ml volumetric flask.

(*b*) Refer to the determination of Sodium Oxide and Potassium Oxide by Flame Photometry, Using the Direct-Intensity Method (ASTM Designation: C 114 - 58 T). Pour 9 ml of the stock $CaCl_2$ solution (63,000 ppm CaO) into the 100-ml flask, and dilute the solution to 100 ml. Determine the sodium and potassium oxide content of this solution as directed. Read the parts per million of each in the solution in the 100-ml flask from the calibration curve.

(*c*) *Calculations.*—Calculate the percentage of water-soluble alkali as Na_2O to the nearest 0.01 as follows:

$$\text{Per cent soluble } Na_2O = \frac{\text{ppm } Na_2O \text{ in solution in 100-ml flask}}{1500}$$

$$\text{Per cent soluble } K_2O = \frac{\text{ppm } K_2O \text{ in solution in 100-ml flask}}{1500}$$

Then, per cent $K_2O \times 0.658$ = per cent Na_2O equivalent of K_2O, and per cent Na_2O + per cent Na_2O equivalent of K_2O = total soluble alkali calculated as Na_2O.

Standard Specifications for Portland Cement (C 150 - 59):[2]

In order to promote uniformity between the various specifications for portland cement, the following revisions are recommended:

Table I.—Delete the limitations on tricalcium silicate for type II and type V cements. Also, place a footnote reference by the value 15 in the column under type III, and add a new footnote *d* to read as follows:

> [d] When moderate sulfate resistance is required for type III cement, tricalcium aluminate may be limited to 8 per cent. When high sulfate resistance is required, the tricalcium aluminate may be limited to 5 per cent.

Table II.—Insert the following requirements for type II cements:

Heat of hydration:[e]	
7 days, maximum cal per g	70
28 days, maximum cal per g	80

In the second sentence of footnote *d*, replace the word "tensile" with the word "compressive." Also, add a new footnote *e* to read as follows:

> [e] These requirements apply only when specifically requested; when the heat of hydration requirements are specified, the strength requirements for type II shall be 80 per cent of the values listed in Table II.

Standard Methods of Test for Autoclave Expansion of Portland Cement (C 151 - 59):[3]

In order to clarify the descriptions of apparatus and test procedures, the following revisions are recommended:

Section 2.—In the third, eighth, and ninth sentences of Paragraph (*c*), replace the words "reference points" with the words "gage studs." Change the seventh sentence to read "Each end plate of the molds shall be equipped to hold properly in place a ¼-in. diameter gage stud of American Iron and Steel Institute type 316 stainless steel."

In Paragraph (*e*), change the first sentence to read "The autoclave shall consist of a high-pressure steam vessel provided with a thermometer well and equipped with an automatic pressure control and a safety valve."

Replace the present Paragraph (*f*) with the following:

> (*f*) *Length Comparator.*—The comparator for measuring length change shall be of such design as to provide or permit the following characteristics (Note 1):
>
> (1) A positive means of contact with the specimen that will ensure reproducible measurements of length,
>
> (2) A high-grade dial micrometer or other measuring device graduated to read in 0.0001-in. units and accurate within 0.0001 in. in any 0.0010-in. range and within 0.0002 in. in any 0.0100-in. range (Note 2),
>
> (3) Sufficient range in the measuring device

to allow for small variations in the gage length of various specimens (Note 3),

(4) Means for checking the measuring device at regular intervals against a standard of reference (Notes 4 and 5), and

(5) Convenient and rapid measurement of specimens.

NOTE 1.—One type of instrument that has been found satisfactory is shown in Fig. 3. In this instrument the contact surfaces are provided by slightly rounded (convex) machined and polished ends on the gage studs in the specimen, and by plane, polished surface heat-treated terminals of the instrument. The terminals shall be fitted with collars $\frac{3}{8}$ to $\frac{1}{2}$ in. long and having an inside diameter 0.02 in. greater than the average diameter of the threaded portion of the gage studs that must fit into the collars. The collars shall be held in place with a set screw.

NOTE 2.—It is recommended that the micrometer or other measuring device be calibrated throughout its range to determine both the periodic and cumulative errors so that proper corrections can be applied to the observed data.

NOTE 3.—If the gage studs are set carefully to position, a dial micrometer with a travel of not less than 0.3 in. provides ample range in the instrument.

NOTE 4.—The reference bar that serves as a standard for the gage dial setting shall have an over-all length of $11\frac{5}{8} \pm \frac{1}{16}$ in. or $6\frac{5}{8} \pm \frac{1}{16}$ in. The bar shall be of a steel alloy having a coefficient of thermal expansion not greater than 1 microinch per in. per deg Fahr temperature. Each end should be machined to the same shape as the contact end of a gage stud, and should be heat treated and hardened and then polished. The central 4 in. of the length of the standard bar should be covered by a rubber tube with a wall at least $\frac{1}{8}$ in. thick to minimize the effect of temperature change during handling. The standard bar should be provided near one end with a positioning mark, and should be placed in the instrument in the same position each time that a length measurement is taken.

NOTE 5.—The dial gage setting of the measuring device should be checked, by use of the reference bar, before and after making the initial and final measurements of each batch of specimens.

Section 4.—Revise Paragraph (*b*) to read as follows:

(*b*) *Preparation of Molds.*—Molds shall be thinly covered with mineral oil; after this operation the gage studs shall be set, care being taken to keep them clean and free of oil.

In the first sentence of Paragraph (*d*), replace the words "reference inserts" with the words "gage studs."

Section 5.—In Paragraph (*a*), revise the first sentence to read "At 24 hr ± 30 min after molding, the specimens shall be removed from the moist atmosphere, immediately measured for length, and placed at once in the autoclave at room temperature in a rack so that all sides of each specimen will be exposed to saturated steam."

In Paragraph (*b*), replace the phrase "1 to $1\frac{1}{4}$ hr" with the phrase "45 to 75 min."

Figures 1 and 2.—In all instances, replace the words "reference points" with the words "gage studs."

Standard Specifications for Air-Entraining Portland Cement (C 175 – 59):[2]

In order to promote uniformity between the various specifications for portland cement, the following revisions are recommended:

Table I.—Delete the limitation on tricalcium silicate for type IIA cements. Also, place a footnote reference by the value 15 in the column under type IIIA, and add a new footnote *b* to read as follows:

[b] When moderate sulfate resistance is required for type IIIA cement, tricalcium aluminate may be limited to 8 per cent. When high sulfate resistance is required, the tricalcium aluminate may be limited to 5 per cent.

Table II.—Insert the following requirements for type IIA cements and a new footnote *d* to read as follows:

Heat of hydration:[d]	
7 days, maximum cal per g	70
28 days, maximum cal per g	80

---

[d] These requirements apply only when specifically requested; when the heat of hydration requirements are specified, the strength requirements for type IIA shall be 80 per cent of the values listed in Table II.

Standard Definitions of Terms Relating to Hydraulic Cement (C 219 – 55):[2]

In order to define more clearly the term "addition," the following revision is recommended:

Addition.—Add a second sentence to read "Two types of Additions are recognized: (1) Processing Additions designed to aid the cement manufacturer in grinding or otherwise processing or handling his cement, and (2) Functional Additions added primarily to modify the end properties of the cement.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 - 58 T),
Flow Table for Use in Tests of Hydraulic Cement (C 230 - 57 T), and
Portland-Pozzolan Cement (C 340 - 58 T).

Tentative Methods of Test for:

Chemical Analysis of Portland Cement (C 114 - 58 T),
Bleeding of Cement Pastes and Mortars (C 243 - 58 T),
Calcium Sulfate in Hydrated Portland Cement Mortar (C 265 - 58 T),
Time of Setting of Hydraulic Cement by Gillmore Needles (C 266 - 58 T),
Flexural Strength of Hydraulic Cement Mortars (C 348 - 57 T), and
False Set of Portland Cement (C 359 - 56 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Upon the recommendation of the Subcommittee on the Cement Reference Laboratory, Committees C-1 on Cement and C-9 on Concrete and Concrete Aggregates have agreed to accept joint responsibility for the Reference Laboratory. It was proposed that the name of the project be changed to the Cement and Concrete Reference Laboratory to reflect the joint sponsorship. These new arrangements were approved by the ASTM Board of Directors at its meeting in January 1960, to become effective in June 1960. The approval of all other interested parties, including the National Bureau of Standards, had been previously obtained.

This report has been submitted to letter ballot of the committee, which consists of 84 voting members; $72\frac{2}{3}$ members returned their ballots, of whom $70\frac{2}{3}$ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. J. MCCOY,
Secretary.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

On Sept. 8, 1960, Committee C-1 on Cement deposited the following reports in the Society's Research Reports file at Headquarters:

File No. RR 5:C-1—Data Considered by Committee C-1 in Preparing the Standard Specifications and Tests for Portland Cement (C 9 - 17); author: Committee C-1 on Cement.

File No. RR 6:C-1—Cooperative Test Program to Investigate the Effect of Mechanical Mixing on the Compressive Strength and Flexural Strength of Portland Cement Mortars (1954); author: C-1 Working Committee on Strength.

File No. RR 7:C-1—Investigation of the Level of Strength Development of Domestic Portland Cement (1954); author: C-1 Working Committee on Strength.

REPORT OF COMMITTEE C-3
ON
CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held two meetings during the year: on Oct. 15 and 16, 1959, in Glens Falls, N. Y.; and on Feb. 25 and 26, 1960, in Cleveland, Ohio.

The committee consists of 28 members, of whom 12 are classified as producers, 8 as consumers, 6 as general interest members, and 2 as consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. R. Allen.
Vice-Chairman, W. H. Burton.
Secretary, E. A. Reineck.

Members of Advisory Subcommittee: Beaumont Thomas, F. H. Buckley, and W. A. Severance.

New Tentative

The committee recommends for publication as tentative the proposed Method of Test for Flexural Strength of Chemically Setting Silicate-Type Chemical-Resistant Mortars, as appended hereto.[1]

Revision of Tentative

The committee recommends revisions as appended hereto[1] of the Tentative Recommended Practice for Use of Hydraulic Cement Mortars in Chemical-Resistant Masonry (**C 398 – 57 T**)[2] and continuation of the recommended practice as tentative.

Adoption of Tentative as Standard with Revision

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Recommended Practice for Installing Sulfur Mortar Joints (C 386 – 59 T):[3]

Revise as appended hereto.[1]

Tentative Specification for Resin-Type Chemical-Resistant Mortars (C 395 – 57 T):[2]

Section 7.—Add a new Paragraph (*e*) to read: "(*e*) *Chemical Resistance.*—Method of Test for Chemical Resistance of Mortars (ASTM Designation: C 267)."

Table I.—Change the title to read: "Physical and Chemical Requirements."

At the end of the first column add "Chemical resistance . . . . ." and place a reference to footnote *a* in each mortar column. Add footnote *a* to read: "[a] Limits to be mutually agreed upon by the consumer and the supplier."

Tentative Method of Test for Compressive Strength of Chemically Setting, Silicate-Type Chemical-Resistant Mortars (C 396 – 57 T):[2]

Section 7 (*c*).—Change the loading

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

[3] 1959 Supplement to Book of ASTM Standards, Part 4.

rate from "600 ± 24 lb per min" to read "6000 ± 500 psi per min."

Tentative Recommended Practice for Use of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 397 - 59 T):[3]

Section 4.—Change to read as follows:

> 4. These mortars may be used with acid-proof brick and tile which must be clean, dry, and warm. Units meeting the requirements of the Specification for Chemical-Resistant Masonry Units (ASTM Designation: C 279) are considered satisfactory. They shall be protected from frost damage, and shall be warmed before using if the temperature is 50 F or below.

Section 8.—Change the last sentence to read: "Silica-filled sodium silicate cement may be used up to 1600 F."

Tentative Recommended Practice for Use of Resin-Type Chemical-Resistant Mortars (C 399 - 59 T):[3]

Table I.—In the column headed "Phenolic" change N to L opposite Alkalies, inorganic; in the column headed "Epoxy" change N to L opposite Bleaches.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Compressive Strength of Resin-Type Chemical-Resistant Mortars (**C 306 - 55**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Section 9 (c).—Change to read: "(*c*) Apply the load to the specimen at the rate of 6000 ± 500 psi per min."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Absorption and Apparent Porosity of Chemical-Resistant Mortars (**C 413 - 58 T**), and

Working and Initial Setting Times of Chemically Setting, Silicate-Type Chemical-Resistant Mortars (**C 414 - 58 T**).

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 28 members; 28 members returned their ballots, of whom 20 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. ALLEN,
Chairman.

E. A. REINECK,
Secretary.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-4

ON

CLAY PIPE*

Committee C-4 on Clay Pipe held one meeting during the year: in Atlantic City, N. J., on June 23, 1959, during the Annual Meeting of the Society.

The committee consists of 28 members of whom 12 are classified as producers, 13 as consumers, and 3 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, C. R. Velzy.
Vice-Chairman, R. H. Welles.
Secretary, R. G. Scott.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specification for:

Extra Strength Clay Pipe (C 200 – 57 T),
Standard Strength Ceramic Glazed or Unglazed Clay Sewer Pipe (C 261 – 57 T), and
Vitrified Clay Pipe Joints Using Materials Having Resilient Properties (C 425 – 58 T).

The standards Committee on Nov. 6, 1959, accepted the revision of the Tentative Specifications C 200 – 59 T and C 261 – 59 T. The revised specifications appear in the 1959 Supplement to the Book of ASTM Standards, Part 5.

Further consideration is being given by Committee C-4 to the revision of the Tentative Specification C 425 – 58 T in light of four negative votes which had been cast in the committee on the revision.

This report has been submitted to letter ballot of the committee, which consists of 28 members; 22 members returned their ballots, of whom 22 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. R. VELZY,
Chairman.

Robert G. Scott,
Secretary.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Standard Strength Ceramic Glazed Clay Pipe (C 462 – 60 T), and
Extra Strength Ceramic Glazed Clay Pipe (C 463 – 60 T).

Revision of Tentative Specifications for:

Standard Strength Ceramic Glazed or Unglazed Clay Sewer Pipe (C 261 – 59 T), with a change in title to read, "Specifications for Standard Strength Unglazed Clay Pipe."

Extra Strength Ceramic Glazed or Unglazed Clay Pipe (C 278 - 57 T), with a change in title to read, "Specifications for Extra Strength Unglazed Clay Pipe."
Vitrified Clay Pipe Joints Using Materials Having Resilient Properties (C 425 - 58 T).

Revision and Reversion to Tentative of Standard Methods of Testing:

Clay Pipe (C 301 - 54).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the new and revised tentative specifications and methods appear in the 1960 Supplement to the Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-7

ON

LIME*

Committee C-7 on Lime held two meetings during the year: on June 24, 1959 in Atlantic City, N. J., during the Annual Meeting of the Society, and on February 3, 1960 in Chicago, Ill., during ASTM Committee Week.

The committee consists of 52 members, of whom 24 are classified as producers, 7 as consumers, and 17 as general interest members, with 4 honorary members.

Committee C-7 regrets the loss of its chairman, Mr. James A. Murray, now deceased. The duties of Mr. Murray were taken over by vice-chairman R. K. Thomas, as Acting Chairman until a new slate of officers is installed.

William J. Barrett has been appointed chairman of Subcommittee IX on Research, due to the resignation of Walter C. Voss.

Subcommittee XI on Agricultural Liming Materials was established, with A. H. Nieman as chairman.

As the result of the work of a Special Task Committee relative to the evaluation of limestone and the study pointing toward a specification for processed limestone for the chemical and process industries, as well as the review of the scope of Subcommittee III, Subcommittee III was charged with the responsibility of determining what specifications may be necessary and desirable and to undertake the preparation and presentation of them.

Subcommittee VII was charged with preparing specifications for hydrated lime for use with pozzolanic materials.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. F. Kriege.

Vice-Chairmen, R. K. Thomas and E. T. Carlson.

Secretary, L. E. Johnson.

TENTATIVE REVISIONS OF STANDARD

The committee recommends tentative revisions as follows of the Standard Methods of Physical Testing of Quicklime and Hydrated Lime (**C 110 - 58**):[1]

New Sections.—Add new Sections 20, 21, and 22 to follow a center heading "Slaking Rate of Quicklime" to read as follows:

SLAKING RATE OF QUICKLIME

20. *Apparatus:*

(*a*) *Mechanical Stirrer,* speed 300 ± 50 rpm (Fisher 14-498 or equivalent) fitted with special stirring rod

(*b*) $\frac{1}{2}$-*qt Modified Dewar Flask* (Fisher 10-197 or equivalent) fitted with special rubber gasket covers

(*c*) *Thermometer,* dial, 0 to 100 C range in 1 C increments

(*d*) *Torsion Balance*

(*e*) *8-in. Sieve, U. S. No. 6*

(*f*) An apparatus essentially the same as that illustrated in Figs. 4 and 5 shall be used. The apparatus consists of a covered reaction container fitted with a mechanical stirrer and thermometer. The quicklime charge shall be stirred with a mechanical stirrer fitted with a glass rod, the end of which is formed into a loop to follow the contour of the reaction container.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 4.

The vacuum reaction flask shall be provided with a cover consisting of two circular pieces of gasket rubber sheet, approximately $\frac{1}{8}$ in. thick. The first piece is provided with a single radial slot which slides over the stirring rod and the thermometer. The second piece (top) has a similar slot plus a hole to provide for the dial thermometer. When the two cover pieces are in place, the slot on the lower piece is at right angles to the slot on the upper piece with the thermometer stem extending through the lower slot. The apparatus may be assembled by any convenient supporting equipment.

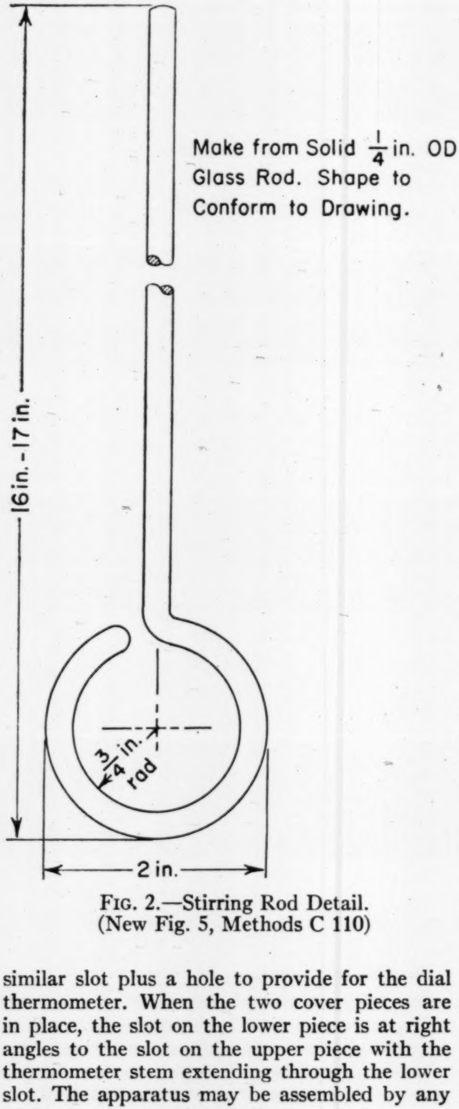

Make from Solid $\frac{1}{4}$ in. OD Glass Rod. Shape to Conform to Drawing.
16 in. - 17 in.
$\frac{3}{4}$ in. rad
2 in.

FIG. 2.—Stirring Rod Detail.
(New Fig. 5, Methods C 110)

21. *Procedure.*—(*a*) Prepare the sample of quicklime to pass a U. S. No. 6 sieve as rapidly as possible to prevent sample deterioration.

(*b*) *Slaking Rate.*—Adjust the temperature of about 500 ml of water in accordance with the schedule below and add the specified amount to the Dewar flask. Set the agitator revolving at 300 ± 50 rpm. The temperature of the water in the flask must be ±0.5 C of the desired temperature. Quarter and weigh out the required amount of the prepared quicklime sample. Add the quicklime to the water without delay. Put the covers in place immediately. Take a reading at 30 sec after adding the quicklime. At this point, note the general reactivity and continue taking readings at the following intervals:

	Material to be Tested	
	Dolomitic	**High Calcium**
Temperature of water, deg Cent.	40	24
Quantity of water, ml	400	380
Quantity of quicklime, g	120	76

Reactivity	**Reading Interval**
High	30 sec
Medium	1 min
Low	5 min

NOTE.—A high reactive lime is generally completely reacted within 10 min; a medium reactive lime is generally completely reacted within 10 to 20 min; and a low reactive lime generally requires more than 20 min for completion.

Continue readings until less than 0.5 temperature change is noted in three consecutive readings. The total active slaking time will then be the time at which the first of the three consecutive readings was taken. The temperature at this time will be considered the final reaction temperature. Subtract the initial temperature from the final temperature to obtain the total temperature rise. Subtract the initial temperature from the temperature at 30 sec for the temperature rise in 30 sec.

22. *Report.*—Record the actual temperature rise and plot a suitable curve showing temperature rise as the ordinate and time as the abscissa. The results may also be reported as:

(*a*) Temperature rise in 30 sec (or at any other designated time) ...... — deg Cent
(*b*) Total temperature rise... — deg Cent
(*c*) Total active slaking time — min

New Figures.—Add new Figs. 4 and

5 as shown in the accompanying Figs. 1 and 2.[2]

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 52 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. K. THOMAS,
Acting Chairman.

L. E. JOHNSON,
Secretary.

---

[2] The new Fig. 4 is not included in this report but appears in the revised methods; see 1960 Supplement to Book of ASTM Standards, Part 4.

[3] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES*

Committee C-8 on Refractories held two meetings during the year: the ninety-sixth at Bedford Springs, Pa., on October 8, 1959; and the ninety-seventh at Chicago, Ill., on February 3, 1960.

The committee consists of 50 members, of whom 27 are classified as producers, 18 as consumers, and 5 as general interest members. The committee takes cognizance of the untimely death of W. R. Kerr, representative of Armstrong Cork Co. who for 8 years served as Secretary of the committee.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. J. Hazel.
Vice-Chairman, S. M. Phelps.
Secretary, W. O. Brandt.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and classifications as appended hereto:[1]

Tentative Method of Test for:

Hydration Resistance of Basic Brick, and Disintegration of Carbon Refractories by Alkali.

Tentative Classification of:

Chrome Brick, Chrome-Magnesite Brick, Magnesite-Chrome Brick, and Magnesite Brick.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

Standard Method of Testing Refractory Brick Under Load at High Temperatures (C 16 – 49):[2]

Section 3(a).—Change to read as follows:

3. (*a*) The test specimen shall consist of a minimum of two 9 by $4\frac{1}{2}$ by $2\frac{1}{2}$- or 3-in. straight refractory bricks, or specimens of this size cut from larger shapes, utilizing as far as possible existing plane surfaces.

Standard Methods of Chemical Analysis of Refractory Materials (C 18 – 52):[2]

Add the following new Sections 79 to 83:

METHOD OF ANALYSIS OF SILICON CARBIDE REFRACTORIES WITH LOW BOND CONTENT

79. *Preparation of the Sample.*—The sample is crushed in a hard tool steel mortar to pass 100-mesh standard screen or equivalent mesh, and thoroughly mixed.

NOTE.—Sample MUST be crushed by impact and not by grinding. A pneumatic hammer and tool steel mortar are preferred.

80. (*a*) *Silicon Carbide Determination.*—Weigh a 1-g sample into a 30-ml platinum crucible; add 15 to 20 ml hydrofluoric acid (48 per cent) and 3 to 4 drops sulfuric acid (sp gr 1.84) and evaporate slowly on a sand bath to volatilize the free silica and silica combined as silicates. Evaporate excess SO_3 fumes, and fuse with approximately 10 g potassium bisulfate at

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 5.

[2] 1958 Book of ASTM Standards, Part 5.

red heat, cool, extract the melt with 75 ml hot water and 20 ml hydrochloric acid (sp gr 1.19). Filter and wash the silicon carbide residue with hot water acidulated with hydrochloric acid. Burn off the filter paper and ignite the silicon carbide residue at about 800 C in a weighed platinum crucible, cool, and weigh.

(*b*) A small amount of silicon carbide is decomposed by the bisulfate fusion and is determined by adding 5 ml sulfuric acid (sp gr 1.84) to the filtrate obtained after filtering off the silicon carbide. Evaporate on a sand bath until fumes of SO_3 appear, in order to dehydrate the silica due to the decomposition of silicon carbide. Cool, dilute with hot water, filter, and wash with hot water acidulated with hydrochloric acid. Burn off the filter paper and ignite to constant weight. The silica is volatilized according to the standard procedure, and is calculated to the equivalent amount of silicon carbide. This is added to the silicon carbide previously weighed. Report as per cent SiC.

81. (*a*) *Calcium Oxide Determination.*—To the filtrate from the silicon carbide determination, add a few drops of nitric acid (sp gr 1.42) and boil to oxidize the iron. Add a few drops of methyl red indicator and carefully add ammonium hydroxide until the color of the solution changes to a distinct yellow. Boil the solution for 1 or 2 min to precipitate the hydroxides of iron, aluminum, and titanium. Filter and wash the precipitate thoroughly with hot ammonium chloride solution (20 g per liter) (be sure the ammonium chloride solution is on the alkaline side). Dissolve the precipitate in dilute hydrochloric acid and reprecipitate, using the same procedure as above.

(*b*) Combine the filtrates from the R_2O_3 precipitation and evaporate to about 200 ml if volume is large. Make the solution slightly ammoniacal and add a few ml of a saturated solution of ammonium oxalate, boil for several minutes, let stand for several hours (preferably overnight), filter and wash with hot water until the washings give no test for oxalate. Dissolve in hot sulfuric acid (1:4) and titrate with 0.05 *N* potassium permanganate at a temperature 70 to 80 F. Report as per cent CaO.

82. *Magnesium Oxide Determination.*—Acidify the filtrate from CaO determination with about 40 ml nitric acid (sp gr 1.42), boil until dry, and heat on hot plate to volatilize the excess ammonium salts. Take up with a few ml of dilute hydrochloric acid, make ammoniacal, filter, and wash. To the filtrate add sodium ammonium phosphate solution (1:10) and then add with constant stirring one tenth of the volume of ammonium hydroxide. Let stand several hours (preferably overnight), filter, and wash with cold ammonium hydroxide solution (1:20). The filter paper and its contents are placed in a weighed platinum crucible and after drying and charring, it is ignited to constant weight. The residue is $Mg_2P_2O_7$; calculate and report as per cent MgO.

83. *Alkalies.*—These are determined by the J. Lawrence Smith method for alkalies in clay, separating potassium as perchlorate.

Standard Definitions of Terms Relating to Refractories (C 71 – 55):[2]

New Definitions.—Under the heading "Materials," add the following new definitions:

Acid Refractories.—Refractories containing a substantial amount of silica which may react chemically, when heated, with basic refractories, basic slags, or basic fluxes.

Basic Refractories.[a]—Refractories whose major constituent is lime, magnesia, or both and which react chemically, when heated, with acid refractories, acid slags, or acid fluxes.

Dead-Burned.—The state of a basic refractory raw material resulting from a heat treatment which yields a product resistant to subsequent atmospheric hydration or recarbonation.

Grain Magnesite.—Dead-burned magnesite in granular form of size suitable for refractory purposes.

Grog.—A granular constituent produced by crushing calcined or burned alumina-silica refractory materials.

Ground Fireclay Mortar.—A refractory mortar consisting of finely ground raw fireclay.

Burn (*n*).—The degree to which desired physical and chemical changes have been developed in refractories during manufacture by heat treatment in a kiln.

Calcine (*n*).—Refractory material, usually fireclay, which has been heated to eliminate volatile constituents and to produce desired volume changes.

Bloating of Refractories.—Swelling of a refractory induced by excessive temperature.

Standard Specifications for Refractories for Incinerators (C 106 – 51):[2]

Section 1.—Change to read as follows:

---

[a] Commercial use of this term also includes refractories made of chrome ore or combinations of chrome ore and dead-burned magnesite.

1. *Scope.*—(*a*) The purpose of these specifications is the selection of refractories for service in municipal and industrial incinerators under general and heavy duty conditions of operation and furnace design.

(*b*) These specifications are intended to cover only burned products made by the usual processes of manufacture.

TABLE I.—REQUIREMENTS FOR FIRECLAY AND SILICON CARBIDE REFRACTORIES.
(Revision of Table I, Specifications C 106)

Tests	Class of Fireclay Refractories						Bonded Silicon Carbide
					Super Duty		
	Low Duty	Medium Duty	High Duty, Spall-Resistant Type	Semi-Silica	Spall-Resistant Type	Slag-Resistant Type	
Pyrometric cone equivalent, min	15	29	31½	...	33	33	...
Panel spalling loss, max	...	...	10 per cent at 2910 F	...	4 per cent at 3000 F	...	...
Hot load subsidence, max	...	...	...	1.5 per cent at 2460 F	...	...	0.2 per cent at 2730 F
Reheat shrinkage, max	...	...	...	...	1 per cent at 2910 F	...	...
Modulus of rupture, min, psi	600	500	500	300	600	1000	...
Bulk density, lb per cu ft, min	...	...	...	...	...	140	155
Porosity, max	...	...	...	...	...	...	18 per cent
Chemical composition	...	...	...	Silica, min, 72 per cent	...	...	SiC, min, 85.0 per cent CaO, max, 0.5 per cent Alkalies, max, 0.25 per cent MgO, max, trace
Number of 9-in. brick required for conducting tests[a]	7	7	21	9	24	7	9

[a] Two extra bricks are included to provide for those which may become damaged in shipment.

NOTE.—When size variation is to be measured in the laboratory, a total of 22 9-in. brick shall be supplied.

Section 2.—Change to read as follows:

2. *Zones of Highest Temperatures.*—Under general conditions of operation and furnace design, the refractories to be used in the zones of highest temperatures, such as the main body of the furnace, crown, and target wall, shall be either spall-resistant high duty or semi-silica fireclay brick. When severe conditions of operations are to be expected, spall-resistant super duty fireclay brick shall be used in these zones. In heavy duty continuous-type incinerators, the refractories to be used in the side walls near the moving grate shall be slag-resistant super duty fireclay brick or bonded silicon carbide brick. The class and type of refractories selected and specified shall conform to the requirements given in Table I.

Section 3.—Replace the last sentence with the following:

Unless spall-resistant high duty or low duty fireclay brick is definitely specified, there shall be supplied for this section of the specifications medium duty fireclay brick. The class and type of refractories supplied or specified shall conform to the requirements given in Table I.

Table I.—Change to read as indicated in the accompanying Table I.

Section 4.—In the first sentence, change "Section 5" to read "Sections 2 and 3."

Section 5.—Delete this section and renumber subsequent sections accordingly.

Section 6.—Renumber as Section 5, and in the first sentence delete the word "fireclay."

Section 8.—Renumber as Section 7. Add the following new Paragraphs (*e*), (*f*), (*h*), (*j*), (*k*), (*l*), and (*m*), deleting the present Paragraph (*e*) and relettering the present Paragraphs (*f*) and (*g*), as (*g*) and (*i*):

(*e*) *Load Test* (2730 F, 1500 C).—Schedule No. 6 of the Method of Testing Refractory Brick Under Load at High Temperatures (ASTM Designation: C 16) except that the 90-min holding period be started at 2730 F.

(*f*) *Reheat Test for Super Duty Brick* (2910 F, 1600 C).—Schedule C of the Method of Test for Reheat Change of Refractory Brick (ASTM Designation: C 113).

(*h*) *Panel Spalling Loss for Super Duty Brick* (3000 F, 1650 C).—Method of Panel Spalling Test for Super Duty Fireclay Brick (ASTM Designation: C 122).

(*j*) *Bulk Density* (Weight per Cubic Foot).—Methods of Test for Size and Bulk Density of Refractory Brick (ASTM Designation: C 134).

(*k*) *Porosity.*—Methods of Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick (ASTM Designation: C 20).

(*l*) *Silica Content.*—Standard Methods of Chemical Analysis of Refractory Materials (ASTM Designation: C 18).

(*m*) *Silicon Carbide Analysis.*—Standard Methods of Chemical Analysis of Refractory Materials (C 18) (Tentative Revision).

Standard Method of Test for True Specific Gravity of Refractory Materials (C 135 – 47):[2]

Section 4.—Change the second sentence of Paragraph (*b*) to read: "The pycnometer shall then be filled with distilled water at room temperature, *t*, which should be between 60 and 75 F and should not vary more than 0.5 F during the test, and again . . . ." In Paragraph (*c*) change "8 to 12 g" to read "approximately 1.5 ml."

Note 2.—Delete the last three sentences.

Section 5.—In Paragraph (*a*) change the equation to read as follows:

$$\text{Sp gr, } t/t = \frac{W - p}{(W_1 - p) - (W_2 - W)}$$

Delete Paragraph (*b*) and Footnote 5.

Section 6.—Change "0.001" to read "0.01" and change "0.005" to read "0.03."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Chemical Analysis of Refractory Materials (**C 18 – 56 T**),[2] and
Thermal Conductivity of Castable Refractories (**C 417 – 58 T**).[2]

Tentative Classification of:

Fireclay and High-Alumina Refractory Brick (**C 27 – 58 T**),[2]
Castable Refractories (**C 401 – 57 T**),[2] and
Silica Refractory Brick (**C 416 – 58 T**).[2]

WITHDRAWAL OF TENTATIVE REVISION

The committee recommends the withdrawal of the tentative revision issued June, 1956, of the Standard Methods of Chemical Analysis of Refractory Materials (**C 18 – 52**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (R. P. Stevens, chairman) is working on revisions to the surveys on the copper industry and on incinerators.

Subcommittee II on Research (R. B. Sosman, chairman) is working on three major problems: (1) the spectrographic analysis of refractories; (2) the study of standard sampling procedure for pyrometric cone equivalent tests, and (3) the permeability of refractories to gases. The subcommittee has completed work on the true relation of the temperature reading to the actual temperature on a temperature gradient in the brick under test in the ASTM hot load test.

Subcommittee III on Tests (S. M. Phelps, chairman) approved the proposed Method of Test for Disintegration of Carbon Refractories by Alkali appended to this report.[1]

Section A on Load (J. D. Sullivan, chairman) is recommending a change in test specimen size to permit the use of 3 in. as well as 2½ in. A task force to study the long-time load test has been appointed.

Section B on Spalling (L. J. Trostel, chairman) has been working on the use of a high-alumina priming coat to be applied to the test brick ahead of the usual kaolin mortar to prevent sticking during the 2910 F and 3000 F preheating. Due to a growing need for a determination of the precision of the spalling test, a program of round-robin testing has been started.

Section C on Temperature (J. L. Carruthers, chairman) reported that the new pyrometric cone equivalent furnace developed at the National Bureau of Standards is finished and tests will start in the near future.

Section D on Precision and Tolerance (W. Duckworth, chairman) has made a study of the formulas for modulus of rupture of nonrectilinear shapes.

Section E on Chemical Analysis (H. A. Heiligman, chairman) is arranging for the preparation and delivery to the National Bureau of Standards of a 200-lb sample of Philippine chrome ore which will be used to replace the previous standard sample of refractory grade chrome ore.

Task Group on Hydration (G. R. Eusner, chairman) is continuing work on methods for measuring the hydration resistance of periclase and dolomite grains.

Section G on Porosity and Permanent Volume Change (L. C. Hewitt, chairman) is working on methods to be used for testing hydratable materials.

Section H on Mortars, Plastics and Castables (G. H. Anthony, chairman) is making a study of the new castable consistency equipment.

Section K on Corrosion Test for Glass Tank Refractories (H. D. Prior, chairman) is at work on the development of various tests for the evaluation of glass tank refractories.

Subcommittee IV on Heat Transfer (C. L. Norton, chairman) is working on methods for testing high conductivity materials such as silicon carbide.

Subcommittee VI on Nomenclature (A. W. Allen, chairman) is revising a number of definitions for refractory materials.

Subcommittee VII on Specifications (J. D. Sullivan, chairman) is revising Specifications C 63, C 64, and C 153.

Section A on Pouring Pit Refractories (J. A. Roslund, chairman) is studying proposed changes in Specifications C 435.

Subcommittee VIII, Editorial (F. W. Schroeder, chairman) reports that all additions and corrections to the present manual have been posted in the master manual.

Subcommittee IX on Classifications (J. A. Kayser, chairman) has prepared a tentative classification of certain basic refractories.

Subcommittee XI on Special Refracto-

ries (A. Maupin, chairman) has prepared a general classification for mullite refractories and is now working on several subclassifications.

Subcommittee XII on Carbon Refractories (E. B. Snyder, chairman) is working on various test methods for testing carbon refractories.

Subcommittee XV on Basic Granular Refractories (J. J. Hazel, chairman) has proposed a tentative classification for dolomite.

This report has been submitted to letter ballot of the committee, which consists of 50 members; 48 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. J. HAZEL,
Chairman.

L. J. TROSTEL,
Secretary.

REPORT OF COMMITTEE C-9
ON
CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held two meetings during the year: on June 24, 1959, during the Annual Meeting of the Society in Atlantic City, N. J., and on Oct. 16, 1959, during the Pacific Area National Meeting of the Society in San Francisco, Calif.

At its June meeting, citations of Mr. A. A. Levison and Mr. L. W. Teller, who had been elected to Honorary Membership in December 1958, were read and copies were presented to them. At its October meeting, the Executive Subcommittee elected Mr. A. D. Conrow to Honorary Membership. Mr. F. B. Lysle was made a Consulting Member.

During the year the committee lost two members through death. Mr. H. F. Thomson, who had represented the American Concrete Institute since 1952, died on Nov. 7, 1959, and Mr. P. D. Miesenhelder, who had represented the Association of American Railroads since 1951, died on Nov. 14, 1959.

The Sanford E. Thompson Award, established by the committee in 1938 in honor of its first chairman, is made for a paper of outstanding merit on concrete or concrete aggregates presented before the Society. The award this year will be made to Messrs. Stanton Walker and Delmar L. Bloem of the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn. for their paper entitled "Variations in Portland Cement," which was presented at the 61st Annual Meeting of the Society, June, 1958, and was published in the 1958 *Proceedings*.

The officers elected for the ensuing term of two years are as follows:

Chairman, Bryant Mather.
Vice-Chairman, Harold Allen.
Secretary, Paul Klieger.

NEW TENTATIVE

The committee recommends for publication as tentative the Recommended Practice for Microscopical Determination of Air-Void Content, Specific Surface, and Spacing Factor of the Air-Void System in Hardened Concrete as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified-Cube Method) (C 116 – 59 T):[2]

Section 1.—In line 1 of the Note, change "method" to read "methods," and add to the text of the note ". . . and (using Simple Beam with Center Point Loading) (ASTM Designation: C 293)." Number the Note as Note 1.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 4.

[2] 1958 Book of ASTM Standards, Part 4.

New Note.—Add to Section 1 the following new Note 2 to read as follows:

NOTE 2.—This method is intended for use in the laboratory and as a research tool for determining relative compressive strength values for various concrete mixtures. It is not intended as an alternate for the Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (ASTM Designation: C 39) and the test values obtained by these two methods are not interchangeable.

Section 3.—Change Paragraph (*a*) to read as follows:

3. (*a*) The bearing faces of the specimen (the sides of the beam as originally molded) shall not depart from a plane by more than 0.002 in.; bearing faces which are not plane shall be ground or capped to meet the 0.002-in. tolerance. Capping procedures shall follow the provisions of Section 10(*c*) of Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (ASTM Designation: C 192). Caps shall cover the full width of the beam and shall be of such length as to permit the adjustment of the bearing plates for the test so that the upper bearing plate may be placed directly over the lower bearing plate.

Add a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as (*c*):

(*b*) *Optional Procedure.*—When specifically requested, beam portions may be tested as modified cubes in the position as originally cast (top of beam up) when otherwise prepared according to Section 3(*a*).

Section 4(*a*).—Revise by adding the following phrase to the second sentence: ". . . and which meet the planeness requirements specified in Section 2 of ASTM Designation C 39."

Section 5.—Revise the first sentence by deleting the words "in pounds per square inch," and substituting "to the nearest 10 psi."

Tentative Method of Test for Volume Change of Cement Mortar and Concrete (C 157 – 54 T):[2]

Change the words "gage plugs" to "gage studs" wherever "gage plugs" appears in the method of test. This change is in line with the definition of terms according to Webster's dictionary. The change would be made in Fig. 1, Section 2 (*a*) (*1*) and (*2*), Notes 1, 3, 5, and 6, and Section 5(*a*) and (*b*).

New Note.—Add a new Note to follow Section 2 (*a*) (*1*) to define more clearly the noncorrosive stainless steel gage stud, to read as follows, renumbering subsequent Notes accordingly:

NOTE 1.—Stainless steel gage studs of some compositions have corroded under the conditions of use required in this test method. Gage studs made of American Iron and Steel Institute type 316 steel alloy have proved satisfactory.

Section 2(*b*).—Change to read as follows, renumbering the Notes as necessary:

(*b*) *Length Comparator.*—The comparator for measuring length change shall be of such design as to provide or permit the following characteristics (Note 1):

(*1*) A positive means of contact with the gage studs that will ensure reproducible measurements of length.

(*2*) A high-grade dial micrometer or other measuring device graduated to read in 0.0001-in. units, and accurate within 0.0001 in. in any 0.0010-in. range, and within 0.002 in. in any 0.100-in. range (Note 2).

(*3*) Sufficient range in the measuring device to allow for small variations in the gage length of various specimens (Note 3).

(*4*) Means for checking the measuring device at regular intervals against a standard of reference (Notes 4 and 5), and

(*5*) Convenient and rapid measurement of specimens.

NOTE 1.—One type of instrument that has been found satisfactory is shown in Fig. 2. In this instrument the contact surfaces are provided by slightly rounded (convex) machined and polished ends on the gage studs in the specimen and by plane, polished surface heat-treated terminals of the instrument. The terminals shall be fitted with collars ⅜ to ½ in. long and having an inside diameter 0.02 in. greater than the average diameter of the threaded portion of the gage studs that must fit into the collars. The collars shall be held in place with a set screw.

NOTE 2.—It is recommended that the micrometer or other measuring device be calibrated throughout its range to determine both the periodic and the cumulative errors, so that proper corrections can be applied to the observed data.

NOTE 3.—If the gage studs are set carefully to position, a dial micrometer with a travel of not less than 0.3 in. provides ample range in the instrument.

NOTE 4.—The reference bar that serves as a standard for the gage dial setting shall have an over-all length of $11\frac{5}{8} \pm \frac{1}{16}$ in. or $6\frac{5}{8} \pm \frac{1}{16}$ in. The bar shall be of a steel alloy having a coefficient of thermal expansion not greater than 1 microinch per in. per deg Fahr. Each end should be machined to the same shape as the contact end of a gage stud, and should be heat-treated and hardened, and then polished. The central 4 in. of the length of the reference bar should be covered by a rubber tube with a wall at least $\frac{1}{8}$ in. thick, to minimize the effect of temperature change during handling. The standard bar should be provided near one end with a positioning mark, and should be placed in the instrument in the same position each time that a length measurement is taken.

NOTE 5.—The dial gage setting of the measuring device should be checked, by use of the reference bar, at least at the beginning and end of readings made within a half day when the apparatus is kept in a room maintained at constant temperature. It should be checked more often when kept in a room where the temperature is not constant.

New Note.—Add the following new Note to Section 5(*b*), numbering as necessary: "NOTE.—When vibration is used, the vibration shall be discontinued as soon as the surface loses its marked irregularities and becomes smooth."

Tentative Specifications for Waterproof Paper for Curing Concrete (C 171 – 59 T):[3]

Section 4(*a*).—Delete the first three lines and that part of line 4 through the word "then" so that the sentence will begin: "Test in . . ."

Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 – 58 T):[2] Revise as appended hereto.[4]

Tentative Method of Testing Air-Entraining Admixtures for Concrete (C 233 – 58 T):[2]

Section 2.—In Paragraph (*a*) change the first and second sentences to read: "The cement used in any series of tests shall be either the cement proposed for specific work in accordance with Section 2(*d*) or a blend of equal parts of three cements. Each cement of the blend shall conform to . . ."

Change the first sentence of Paragraph (*b*) to read: "Except when tests are made in accordance with Section 2 (*d*) using the aggregates proposed for specific work, the fine and coarse aggregates used . . ."

To Paragraph (*d*) add a second sentence to read "If the maximum size of coarse aggregate is greater than 1 in., the freshly-mixed concrete shall be screened over a 1-in. sieve prior to fabricating the test specimen."

Section 3.—Substitute the following for the existing Paragraph (*a*) and paragraph heading:

(*a*) *Concrete Mixtures.—Proportions.*—Using the ACI Standard Recommended Practice for Selecting Proportions for Concrete (ACI 613-54), all concrete shall be proportioned to conform to the following requirements:

(*1*) The cement content shall be 5.5 ± 0.05 bags per cu yd, except when tests are being made for specific uses (Section 2 (*d*)).

(*2*) The first trial mixture shall contain the amount of coarse aggregate shown in Table 6 of ACI 613-54 for the maximum size of aggregate and for the fineness modulus of the sand being used.

(*3*) For the nonair-entrained mixtures, the air content used in the computation of proportions shall be 1.5, as shown in Table 3 of ACI 613-54. For the air-entrained mixtures, the air content used for this purpose shall be the middle of the range of air content shown in (*b*) of this paragraph.

(*4*) The water content and sand content shall be adjusted to obtain a slump of $2\frac{1}{2} \pm \frac{1}{2}$ in. The workability of the concrete mixture shall be suitable for consolidation by hand rodding and the concrete mixture shall have the minimum water content possible. These conditions shall be achieved by final adjustments in the proportion of fine aggregate to total aggregate, in the amount of total aggregate, or both, while maintaining the yield and slump in the required ranges.

---

[3] 1959 Supplement to Book of ASTM Standards, Part 4.

[4] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 4.

Change Paragraph (*b*) to read as follows:

(*b*) Concrete mixtures shall be prepared both with and without the air-entraining admixture under test and with the reference admixture. The admixtures shall be added in the amounts necessary to produce 5.5 ± 0.5 per cent of air in the concrete, except where the admixture under test is for use in specific work (see Section 2 (*d*)). In this case the amount of admixture added shall be sufficient to produce concrete of the air content specified for use in the work.

Section 4.—Change the first sentence to read: "Concrete shall be machine-mixed in accordance with Section 4(*c*) of ASTM Method C 192."

Section 6.—To the end of the first sentence of Paragraph (*a*) add the words "except that for freezing-and-thawing tests a minimum of 6 specimens shall be used instead of 3." Replace the last two sentences with the following:

Each group of specimens representing a given test at a given age shall be examined visually before they are tested. Any specimen found to be manifestly faulty by such examination shall be discarded without being tested. All specimens representing a given test at a given age shall be examined visually after testing, and the results obtained from any found manifestly faulty by such examination shall be discarded. Should more than one specimen representing a given test at a given age be found manifestly faulty either before or after testing, the entire test shall be discarded and repeated. The test results reported shall be the average of the individual test results of the specimens tested or, in the event that one specimen or one result has been discarded, the average of the remaining specimens.

In Paragraph (*b*), Item (*4*) delete the words "... reinforcing bars of $\frac{3}{4}$ in. nominal diameter ..." The present method of test specifies the size of the bar.

Section 7(d).—Change the second sentence to read as follows:

The strength of the bond shall be evaluated in terms of the nominal average bond stress measured at five equal intervals of slip at the loaded end taken from the bond stress curves, over the range of zero to 0.01-in. slip. If the maximum slip for any specimen, or specimens, in the sets being compared is less than 0.01 in., then the upper limit of the range over which the comparisons are to be made shall be the lowest maximum slip in the group.

Table I.—In the right-hand column, sixth row down, change the present "6" to read "12."

Tentative Specifications for Air-Entraining Admixtures for Concrete (C 260 – 58 T):[2]

Section 3(d).—Substitute the following for the present definitions of *DF* (or *DF*₁), *P*, and *N*:

$$DF(\text{or } DF_1) = \frac{PN}{300}$$

P = relative dynamic modulus of elasticity in percentage of the dynamic modulus of elasticity at zero cycles (values of P will be 60 or greater).

N = number of cycles at which P reaches 60 per cent, or 300 if P does not reach 60 per cent prior to the end of test (300 cycles), and ..."

This change is to bring the test in line with Method C 290.

Tentative Specifications for Lightweight Aggregates for Structural Concrete C 330 – 59 T):[3]

Section 1.—Add a new sentence to read: "Procedures outlined in this specification are not intended for job control of concrete."

Change line 1 of the Note to read "Aggregates for use in concrete for such purposes as fire- ... "

Tentative Specifications for Fly Ash for Use as an Admixture in Portland-Cement Concrete (C 350 – 57 T):[2]

Revise as appended hereto.[4]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Unit Weight of Aggregate (**C 29 – 55 T**),[2]
Organic Impurities in Sands for Concrete (**C 40 – 56 T**),[2]
Air Content of Freshly Mixed Concrete by the Pressure Method (**C 231 – 56 T**),[2] and
Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (**C 215 – 58 T**).[2]

Tentative Specification for:

Packaged, Dry, Combined Materials for Mortar and Concrete (**C 387 – 58 T**).[2]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Securing, Preparing, and Testing Specimens from Hardened Concrete for Compressive and Flexural Strengths (**C 42 – 59**)[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 7.—Change Paragraph (*d*) to read as follows:

> (*d*) *Measurement.*—Prior to testing, the length of the capped specimen shall be measured to the nearest 0.1 in. and its average diameter determined from two measurements taken at right angles to each other at about midheight of the specimen. Shot-drilled core diameters shall be measured within an accuracy of 0.10 in. Diamond-drilled core diameters shall be measured to the nearest 0.01 in.

In Paragraph (*f*) change the second sentence to read: "The compressive strength of each specimen shall be calculated to the nearest 10 psi based on the average diameter of the specimen."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Measuring Mortar-Making Properties of Fine Aggregate (**C 87 – 58 T**),
Clay Lumps in Natural Aggregates (**C 142 – 55 T**),
Water Retention Efficiency of Liquid Membrane-Forming and Impermeable Sheet Materials for Curing Concrete (**C 156 – 55 T**),
Comparing Concretes on the Basis of the Bond Developed with Reinforcing Steel (**C 234 – 57 T**),
Scratch Hardness of Coarse Aggregate Particles (**C 235 – 57 T**),
Potential Reactivity of Aggregates (Chemical Method) (**C 289 – 57 T**),
Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (**C 290 – 57 T**),
Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (**C 291 – 57 T**),
Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (**C 292 – 57 T**),
Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (**C 310 – 57 T**),
Potential Volume Change of Cement-Aggregate Combinations (**C 342 – 55 T**),
Ball Penetration in Fresh Portland-Cement Concrete (**C 360 – 55 T**),
Rate of Hardening of Mortars Sieved from Concrete Mixtures by Proctor Penetration Resistance Needles (**C 403 – 57 T**), and
Abrasion Resistance of Concrete (**C 418 – 58 T**).

Tentative Specifications for:

Lightweight Aggregates for Insulating Concrete (**C 332 – 56 T**), and
Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland-Cement Concrete (**C 402 – 58 T**).

EDITORIAL REVISIONS

The committee recommends editorial changes as indicated in the following standards:

Tentative Method of Test for Lightweight Pieces in Aggregate (C 123 – 57 T):[2]

Section 3.—In Paragraph (*a*), line 3, and Paragraph (*b*), tabulation, line 1, change "acetylene tetrabromide" to read "1,1,2,2,tetrabromoethane."

Standard Method of Test for Bleeding of Concrete (C 232 – 58):[2]

Section 4.—In line 7, change "on" to read "or."

Tentative Methods of Sampling and Testing Fly Ash for Use as an Admixture in Portland-Cement Concrete (C 311 – 59 T):[3]

Section 13.—In line 11, place a period after C 114 and delete the remainder of the sentence.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

This report has been submitted to letter ballot of the committee, which consists of 187 active members commanding 139 votes plus 8 honorary members having voting privileges; 166 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. PRICE,
Chairman.

BRYANT MATHER,
Secretary.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-11

ON

GYPSUM*

Committee C-11 on Gypsum held two meetings during the year: in Atlantic City, N. J., on June 25 and 26, 1959, and in Chicago, Ill., on Feb. 1 and 2, 1960.

The committee consists of 30 members, of whom 29 are voting members; 12 are classified as producers, 8 as consumers, 9 as general interest members, with one consulting member.

The officers elected for the ensuing term of two years are as follows:

Chairman, G. W. Josephson.
Vice-Chairman, B. W. Nies.
Secretary, R. H. Faber.
Executive Committee: Max Barth, T. I. Coe, E. T. Carlson, and O. H. Storey, Jr.

Revision of Tentative

The committee recommends revisions as follows of the Tentative Specifications for Annular Ringed Nails for Gypsum Wallboard (**C 380 - 58 T**)[1] and continuation of the specifications as tentative:

Section 4.—Change to read as follows:

Length, in.	Approximate Number per Pound
1⅛	375
1¼	350
1⅜	325

Revision of Standards, Immediate Adoption

The committee recommends for immediate adoption revision as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Definitions of Terms Relating to Gypsum (C 11 - 59):[2]

New Definition.—Add a definition for Gypsum Backing Board to read as follows:

Gypsum Backing Board.—A sheet or slab having an incombustible core, essentially gypsum, surfaced with paper on both sides and designed to be used as a base layer, or a backing material, in multilayer construction.

Standard Methods of Testing Gypsum and Gypsum Products (C 26 - 59):[2]

Section 3.—Renumber as Section 18, renumbering all sections between the present Sections 4 and 18.

Section 10.—Change the third, fourth, and fifth sentences to read as follows: "Stir vigorously until precipitate begins to form. Let stand overnight. Filter using a Gooch crucible, wash with . . . ."

Section 18.—Add a new footnote referenced to the title of this section, the footnote to read "For mixing pastes and mortars a 500-ml rubber dental bowl is a convenience."

To Paragraph (*b*) add the following: "for mixing pastes and mortars. Tap water may be used in the tests for fineness, wood fiber, and water resistance."

In Paragraph (*c*), in the last sentence delete the words "once with HCl (1:4) and." Add a new sentence to read: "Store in closed containers."

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 4.

[2] 1959 Supplement to Book of ASTM Standards, Part 4.

Section 24.—In Paragraph (*a*) change the first sentence to read as follows: "Starting the timing of the test approximately at the moment of contact of the dry material with the water, mix 200 g of the sample . . . ." In the third sentence delete the words "at frequent intervals."

In Paragraph (*b*) change the first sentence to read "Test gypsum neat plaster for time of setting as mixed with three parts by weight of standard sand as described in Section 18 (*c*)." Delete the last three sentences of this paragraph.

Add a new Paragraph (*c*), Frequency of Testing, to consist of the following chart:

Kind of Material	First Test	Frequency of Subsequent Tests
Molding plaster. . . .	15 min	5 min
Keene's cement:		
Standard.	15 min	1 hr
Quick set.	15 min	5 min
Gypsum concrete. . .	15 min	5 min
Ready mixed plaster.	1½ hr	1 hr
Neat plaster.	2 hr	1 hr
Wood fibered.	1½ hr	1 hr
Bond plaster.	1½ hr	1 hr
Gaging plaster:		
Slow set.	40 min	2 hr
Quick set.	15 min	5 min

Section 26.—Change the last sentence of Paragraph (*a*) to read "For gypsum concrete, soak for 1 min and stir vigorously (about 150 complete circular strokes per minute) with a large metal spoon for 3 min."

In Paragraph (*b*) delete the last three sentences and replace with the following: "Place the cubes in an oven provided with air circulation and adequate ventilation for removal of moisture, so that the air may be maintained at a temperature of 90 to 110 F, and a relative humidity not to exceed 50 per cent. Dry the cubes to constant weight as determined by weighing once each day but not to exceed 7 days, and then place in a desiccator over magnesium perchlorate for 24 hr before testing. Cubes shall be tested as soon as removed from the desiccator."

Section 28.—Delete the Note on Tensile Strength Tests following this section.

Section 37.—In the eighth line, following the words "70 to 100 F" insert "(21 to 38 C)."

Section 41.—In Paragraph (*b*), in the first line delete the words "self-reading."

Section 47.—In the first and fourth lines delete the words "self-reading."

Appendix.—In the second paragraph, in the fourth and fifth lines change "an accurately weighed 50-g" to read "50 ± 0.1 g." In the fifth line of the second column change "for exactly 2 min" to read "for 2 min ± 2 sec."

Standard Specifications for Gypsum Plasters (C 28 - 59):[2]

New Note.—Add a new Note between Sections 2 and 3 and immediately following the center heading "Chemical and Physical Properties" to read as follows:

NOTE.—*Time of Setting.*—Attention is directed to conditions affecting the job set of gypsum plasters which are usually beyond the control of the producer. Materials added at the site of application such as water and aggregates affect job sets. In addition, the various bases with different absorptive values may affect job performance. Attention is directed to the requirements of American Standard Specifications for Gypsum Plastering, A42.1, Sections 10.10 and 10.20, Application of Basecoats, the last sentence of which states "Slow setting plaster shall be accelerated to provide a set (mixed material) of less than 4 hr."

Standard Specifications for Gypsum Wallboard (C 36 - 58):[1]

Section 5.—In Paragraph (*a*), in the last sentence change the words "at least 0.010 in. less than" to read "at least

0.010 in. but not more than 0.060 in. less than."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 29 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. W. JOSEPHSON,
Chairman.

R. H. FABER,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE*

Committee C-13 on Concrete Pipe held one meeting during the year: in San Francisco, Calif., on Oct. 16, 1959.

The committee consists of 41 members, of whom 18 are classified as producers, 13 as consumers, and 10 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. R. Litehiser.
Vice-Chairman, E. F. Bespalow.
Secretary, Howard F. Peckworth.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Reinforced Concrete Culvert, Storm Drain and Sewer Pipe (C 76 – 59 T):[1]

Section 11.—Change Paragraph (*a*) to read as follows:

> 11. (*a*) Placing Reinforcement.—Where one line of circular reinforcement is used, it shall be placed from 35 to 50 per cent of the shell thickness from the inner surface of the pipe except that for wall thicknesses less than $2\frac{1}{2}$ in., the nominal protective cover of the concrete over the circumferential reinforcement in the barrel of the pipe shall be $\frac{3}{4}$ in. In circular pipe having two lines of circular reinforcement, each line shall be so placed that the nominal protective covering of concrete over the circumferential reinforcement in the barrel of the pipe shall be 1 in. In circular pipe having elliptical reinforcement, and in elliptical pipe having circular reinforcement with wall thicknesses $2\frac{1}{2}$ in. or over, the reinforcement in the barrel of the pipe shall be so placed that the nominal covering of concrete over the circumferential reinforcement along the vertical diameter of the pipe shall be 1 in. from the inside surface of the pipe, and the nominal protective covering of concrete over the circumferential reinforcement along the horizontal diameter of the pipe shall be 1 in. from the outside surface of the pipe. In all pipe 36 in. or more in diameter, the bell or the spigot of the joint shall contain circumferential reinforcement equal in area to that of a single line within the barrel of the pipe. The nominal location of the reinforcement shall be subject, however, to the permissible variations in dimensions given in Section 25.

Change the first sentence of Paragraph (*b*) to read: "(*b*) A line of circumferential reinforcement of any given total area . . . ."

Section 25(b).—Change the last sentence to read: "In no case, however, shall the cover over the circumferential reinforcement be less than $\frac{1}{2}$ in. for wall thicknesses less than $2\frac{1}{2}$ in., or less than $\frac{3}{4}$ in. for wall thicknesses $2\frac{1}{2}$ in. or greater."

Tables III, IV and V.—Adjust Wall C minimum wall thickness so that they are equal to $D/12 + 1\frac{3}{4}$ in. Also, change the third paragraph under the note at the top of each table to read: "The minimum compressive strength of the concrete at the time of acceptance under Section 3(*a*)(*2*) or 3(*a*)(*3*) shall be as shown in this table."

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 5.

Tentative Specifications for Joints for Circular Concrete Sewer and Culvert Pipe, Using Flexible, Watertight, Rubber-Type Gaskets (C 443 - 59 T):[2]

Section 1(b).—Add a sentence to the Note following this paragraph to read: "The purchaser may specify other infiltration or exfiltration requirements."

Section 4.—In the tabular material change the reference to accelerated aging to read as follows: "Accelerated Aging.—To be conditioned in a circulating hot air oven 96 hr at 158 F. Tensile strength loss, 15 per cent max; elongation loss, 20 per cent max per cent of original." Change the water absorption requirement from "5 per cent" to "10 per cent."

Section 5(f).—Change the first sentence to read: "(*f*) The annular space with the joint centered, including the manufacturer's . . . ."

Section 6(c).—Change to read as follows:

> (*c*) The manufacturer's tolerances for the width of the annular space between the gasket bearing surfaces shall not vary by more than ±10 per cent of the uncompressed thickness of the applied gasket.

Section 7(e).—Change to read: "(*e*) *Accelerated Aging.*—Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (D 573)."

Tentative Specification for Perforated Concrete Pipe (C 444 - 59 T):[2]

Section 11.—Change the first sentence to read: "Perforations shall be circular, not greater than $\frac{5}{16}$ in. nor less than $\frac{3}{16}$ in. in diameter and arranged in rows parallel to the axis of the pipe."

Section 19.—Add a new Paragraph (*j*) to read: "(*j*) Any spall greater than $\frac{3}{4}$ in. in diameter and $\frac{3}{16}$ in. in depth caused by making perforations."

Table II.—Delete the words "Bell and Spigot" from the table title.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Concrete Drain Tile (**C 412 - 58 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision of Section 5 of the Standard Specifications for Concrete Pipe for Irrigation or Drainage (**C 118 - 59**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee which consists of 41 voting members; 36 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

HOWARD F. PECKWORTH,
Secretary.

---

[2] 1959 Supplement to Book of ASTM Standards, Part 5.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-15
ON
MANUFACTURED MASONRY UNITS*

Committee C-15 on Manufactured Masonry Units held two meetings during the year: on June 26, 1959, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and on Feb. 4, 1960, in Chicago, Ill., during ASTM Committee Week.

The committee, in cooperation with Committee C-12 on Mortars for Unit Masonry, sponsored a Symposium on Masonry Materials at the Third Pacific Area National Meeting held in San Francisco, Calif., on October 12, 1959.

The committee consists of 73 members, of whom 36 are classified as producers, 17 as consumers, and 20 as general interest members. It is now in exact balance with respect to the number of producer members as compared to the total of consumer and general interest members.

During the past year the committee lost one of its most valuable members of long standing: Dalton G. Miller, Honorary Member, and for many years Chairman of the Subcommittee on Drain Tile, passed away on June 26, 1959, after a long illness.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. W. Whittemore.

First Vice-Chairman, P. M. Woodworth.

Second Vice-Chairman, J. A. Lee.

Secretary, M. H. Allen.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard:

Methods of Sampling and Testing Brick (C 67 - 57): Sections 9, 11(a) and 11(c).

Specifications for Facing Brick (C 216 - 57): Section 4(a) and Explanatory Note 2.

Revision of Tentative Revision of Standard:

Specifications for Building Brick (C 62 - 58): Explanatory Note C and Fig. 1.

Revision of Tentative Specifications for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick and Solid Masonry Units (C 126 - 58 T): Section 5(a), and Tables III and V.

Revision and Reversion to Tentative of Standard Specifications for:

Vitrified Clay Filter Blocks for Trickling Filters (C 159 - 55).

These recommendations were accepted by the Standards Committee on November 6, 1959, and the revisions appear in the 1959 Supplement to Book of ASTM Standards, Part 5.

---

* **Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.**

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions, as indicated, of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Standard Methods of Sampling and Testing Brick (**C 67 – 57**):[1] Section 10, issued September, 1956.

Standard Methods of Sampling and Testing Structural Clay Tile (**C 112 – 52**):[1] Section 10, issued November, 1957.

Standard Specifications for Structural Clay Facing Tile (**C 212 – 54**):[1] Section 4(e), issued November, 1957.

Standard Specifications for Facing Brick (**C 216 – 57**):[1] Sections 3, 4 and 7, issued November, 1957.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Specifications for Industrial Floor Brick (**C 410 – 57 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Volume Change of Concrete Products (**C 341 – 54 T**),[1]

Drying Shrinkage of Concrete Block (**C 426 – 58 T**),[1] and

Moisture Condition of Hardened Concrete by the Relative Humidity Method (**C 427 – 58 T**).[1]

TENTATIVE REVISIONS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the tentative revisions as indicated of the following standards:

Standard Specifications for:

Building Brick (**C 62 – 58**):[1] Sections 1(*a*) and 2(*f*), and Explanatory Note C, issued September, 1956.

Facing Brick (**C 216 – 57**):[1] Section 6(a), and Explanatory Note 8, issued September, 1958.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Executive (J. W. Whittemore, chairman).—Problems concerning membership was the item of major concern during the year. Through a stricter enforcement of attendance and letter ballot requirements, resulting in the dropping of several inactive members and the addition of a number of new members representing consumer and general interest elements, a membership balance in accordance with Society Regulations was achieved.

The Executive Subcommittee is giving consideration to the possibility of Committee C-15 sponsoring a research fellowship.

Subcommittee II on Clay Brick and Structural Clay Tile (J. A. Lee, chairman) has continued its active review of the numerous specifications and methods of test under its jurisdiction and has recommended a number of tentative revisions which have been approved by the committee and the Society. In addition, several task groups have been working on specific studies, the results of which will form the basis for further desirable revisions which will either simplify or upgrade the specifications.

Subcommittee III on Concrete and Sand Lime Units (R. E. Copeland, chairman) has had under discussion a number of desirable revisions of standards and tentatives under its jurisdiction which are awaiting approval or agreement among industry groups. The Technical Problems Committee of the National Concrete Masonry Association is currently preparing a number of such revisions which will be presented for consideration in the very near future.

---

[1] 1958 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Water-Retardant Materials for Masonry Units (C. C. Connor, chairman) has under way a cooperative testing program, the data from which it is hoped will be helpful in developing a standard method of test for water vapor transpiration through water-retardant materials.

Another task group is conducting tests looking toward the development of a standard method of test for measuring the effectiveness of transparent water-retardant materials.

A third group has started a series of tests for the development of test methods for measuring the effectiveness of cementitious-type paints as water retardants.

Subcommittee VI on Glazed Brick and Tile (E. F. Wanner, chairman) has recommended a number of revisions of the Tentative Specifications for Ceramic Glazed Structural Clay Facing Tile (C 129) to tighten the permissible tolerances on dimensions and distortion and, in general, to upgrade the specifications. The subcommittee hopes to recommend that this tentative be adopted as standard some time during the ensuing year. Because of the necessity for constant revision due to new developments in this industry, this specification had been kept in a tentative status for a number of years.

Subcommittee VIII on Clay Filter Block (E. F. Wanner, chairman) completed its revision of the Standard Specifications for Vitrified Clay Filter Block for Trickling Filters (C 159) and recommended that the specification be reverted to a tentative status.

Subcommittee IX on Chemical-Resistant Units (W. W. Perkins, chairman.—Mr. V. W. Weidman, chairman of this subcommittee since its formation over 11 years ago, found it necessary to resign during 1959. The chairmanship was taken over very capably by W. W. Perkins, also a long-time member of the subcommittee.

The subcommittee continued its work on the development of a specification for chemical-resistant ceramic tower packings. A series of round-robin tests is being conducted to obtain data which will help in determining strength limits and in the development of standard test methods. The subcommittee hopes to be able to make specific recommendations relative to such a proposed specification within the next year.

Subcommittee XI, Editorial (F. A. Biberstein, chairman) is now more active than it has been for many years. The expanded subcommittee is carefully reviewing all standards and tentatives under the jurisdiction of Committee C-15 and is transmitting to the proper subcommittee chairmen its recommendations relative to desirable editorial changes in the interest of maintaining uniformity of presentation, compliance with ASTM style and format, and clarity of expression.

This report has been submitted to letter ballot of the committee, which consists of 73 members; 57 members returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
Chairman.

M. H. Allen,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 – 59 T).

Tentative Revision of Standard Specifications for:

Building Brick (C 62 – 58).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the revised tentative specifications and the tentative revision of standard appear in the 1960 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-16
ON
THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the year: at Pocono Manor, Pa., on Sept. 27–30, 1959, and at Charleston, S. C., on March 8–11, 1960.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. C. Lewis.
First Vice-Chairman, J. Roy Allen.
Second Vice-Chairman, C. B. Bradley.
Secretary, J. M. High.
Assistant Secretary, J. B. Mochel.
Membership Secretary, E. C. Shuman.
Member-at-Large-to Executive Committee, W. L. Scott.

During the year, the committee was saddened by the death of one of its members, Harry E. Lewis of M. H. Dietrick Co. who died July 20, 1959. Mr. Lewis had been an untiring worker on the committee for many years.

At the Pocono Meeting the immediate past chairman, William L. Gantz, who was required to resign because of a change in his position, was honored with the presentation of a scroll commending him on his work in aiding and forwarding the purposes and progress of Committee C-16.

The committee consists of 100 voting members, of whom 48 are classified as producers, 28 as consumers, and 24 as general interest members. There are also 24 non-voting members, 8 associate members, and 5 honorary members.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1959 Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations which were accepted on the dates indicated:

New Tentative Recommended Practice for:

Prefabrication or Field Fabrication of Thermal Insulating Fitting Covers (**C 450 – 60 T**) (accepted March 23, 1960).

New Tentative Specification for:

Mineral Fiber Hydraulic Setting Thermal Insulating and Finishing Cement (**C 449 – 60 T**) (accepted February 29, 1960).

Revision of Tentative Method of Test for:

Mean Specific Heat of Thermal Insulation (**C 351 – 59 T**) (accepted September 15, 1959).

Revision of Tentative Specification for:

Calcium Silicate Block-Type Thermal Insulation (**C 344 – 54 T**) (accepted March 23, 1960),

Calcium Silicate Thermal Insulation for Pipes (**C 345 – 54 T**) (accepted March 23, 1960), and

Mineral Wool Block or Board Thermal Insulation for Low Temperatures (**C 378 – 56 T**) (accepted March 23, 1960).

The tentative recommended practice includes the issuance of a separate manual of dimensions covering all the sizes and styles of covers involved.

---

* **Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.**

The new tentative specification, recommended practice, and revisions will appear in the 1960 Supplement to the Book of ASTM Standards, Part 5.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of the Standard Definitions of Terms Relating to Thermal Insulating Materials (**C 168 – 56**):[1]

New Definition.—Add the following new definition:

Mineral Fiber Blanket Thermal Insulation.—A blanket thermal insulation composed of inorganic fibers, with or without added binders.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specification for:

Structural Insulating Board Made From Vegetable Fibers (**C 208 – 57 T**),[1] and
Mineral Wool Block or Board Thermal Insulation for Elevated Temperatures (**C 392 – 57 T**).[1]

Tentative Method of Test for:

Thermal Conductance and Transmittance of Built-Up Sections by Means of the Guarded Hot Box (**C 236 – 54 T**),[1]
Linear Shrinkage of Preformed High-Temperature Thermal Insulation Subjected to Soaking Heat (**C 356 – 55 T**),[1] and
Consistency of Wet-Mixed Thermal Insulating Cement (**C 405 – 57 T**).[1]

Tentative Method of:

Sampling Preformed Thermal Insulation (**C 390 – 57 T**).[1]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Testing Structural Insulating Board Made from Vegetable Fibers (**C 209 – 57 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard with the following revisions:

Section 31.—Insert the words "Desiccant Method" so that the first sentence in this section will read, "Determine vapor transmission in accordance with the Methods of Test for Water Vapor Transmission of Materials Used in Building Construction (ASTM Designation: C 355, Desiccant Method)."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specification for:

Mineral Wool Batt Insulation (Industrial Type) (**C 262 – 55 T**),
Mineral Wool Felt Insulation (Industrial Type) for Low Temperatures (**C 264 – 55 T**),
Mineral Wool Molded-Type Pipe Insulation for Elevated Temperatures (**C 281 – 52 T**),
Mineral Wool Molded-Type Pipe Insulation for Low Temperatures (**C 300 – 52 T**), and
Mineral Wool Felt Insulation (Industrial Type) for Elevated Temperatures (**C 382 – 56 T**).

Tentative Method of Test for:

Thermal Conductivity of Pipe Insulation (**C 335 – 54 T**),
Mean Specific Heat of Thermal Insulation (**C 351 – 54 T**),
Thermal Conductivity of Insulating Materials at Low Temperatures by Means of the Wilkes Calorimeter (**C 420 – 58 T**), and
Mechanical Stability of Preformed Thermal Insulation by Tumbling (**C 421 – 58 T**).

Tentative Method of:

Making and Curing Test Specimens of Mastic Thermal Insulation Coatings (**C 419 – 58 T**).

EDITORIAL CHANGES

Replace the words "mineral wool" with "mineral fiber" in the title and text of all standards and tentatives for mineral wool thermal insulating materials under the jurisdiction of Committee C-16.

---

[1] **1958 Book of ASTM Standards, Part 5.**

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-III on Insulating Cement (E. F. Karklin, chairman).—A new scope for the subcommittee was adopted as follows:

Develop and maintain specifications and test methods applicable to cements associated with thermal insulation.

Subcommittee S-VIII on General Standards (H. P. Hoopes, chairman).—In addition to the task of developing a recommended practice for prefabrication and field fabrication of thermal insulating fitting covers with the supporting manual of dimensions for styles and shapes, the subcommittee is currently working on recommended practices for (1) cutting beveled blocks on preformed thermal insulation, (2) outside pipe insulation diameters, and (3) squareness and trueness of block and pipe insulation.

Subcommittee S-XI on Reflective Insulation (G. E. Gronemeyer, chairman) has studied and is proposing a research program on reflective insulations used within gaseous environments at high temperatures and high pressures. Favorable action was taken by Committee C-16 subject to committee letter ballot.

This report has been submitted to letter ballot of the committee, which consists of 100 voting members; 85 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. C. LEWIS,
Chairman.

J. M. HIGH,
Secretary.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Mastic Coatings Used in Connection with Insulations (C 461 - 60 T).

Revision of Tentative Specifications for:

Mineral Fiber Blanket and Felt Insulation (Industrial Type) for Low Temperatures (C 264 - 55 T).

Revision and Reversion to Tentative of Standard Specifications for:

Mineral Fiber Thermal Insulating Cement (C 195 - 48).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the new and revised tentative method and specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-17
ON
ASBESTOS-CEMENT PRODUCTS*

Committee C-17 on Asbestos-Cement Products and its subcommittees met on June 25, 1959, at Atlantic City, N. J., during the Annual Meeting of the Society and on Feb. 17 and 18, 1960, in New York City.

The committee consists of 38 members, of whom 33 are voting members; 10 are classified as producers, 4 as consumers, 17 as general interest members, and 2 as consulting members. G. W. Francis, of The Francis Engineering Co., Saginaw, Mich., was elected to membership. Plans have been formulated to obtain additional members, especially consumer members. S. E. Williams was reappointed as liaison member on Committee E-6 on Methods of Testing Building Constructions.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. V. Friedlaender.

Vice-Chairman, C. R. Hutchcroft.

Secretary, S. Collier.

N. B. Jackson was appointed as Assistant Secretary.

Reorganization of Subcommittees.—In order to facilitate the increased activity in Committee C-17, the subcommittee structure was reorganized as follows:

Subcommittee I, Nomenclature and Editorial (W. A. Heausler, chairman)

Subcommittee II on Research (William Lerch, chairman)

Subcommittee III, ISO/TC 77 Asbestos-Cement Products (J. R. Cran, chairman)

Subcommittee IV on Roofing and Siding (F. Crippen, chairman)

Subcommittee V on Flat and Corrugated Board (S. E. Williams, chairman)

Subcommittee VI on Pipe (C. R. Meek, chairman)

ISO/TC 77 on Asbestos-Cement Products.—R. F. Orth was recommended to be the United States delegate to the next meeting of ISO/TC 77 to be held in Stockholm, Sweden, on June 13 to 17, 1960.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and definitions as appended hereto:[1]

Tentative Methods of:

Test for Organic Fiber Content of Asbestos-Cement Products, and

Sampling and Testing Asbestos-Cement Flat Sheets, Roofing and Siding Shingles, and Clapboards.

Tentative Definitions of:

Terms Relating to Asbestos-Cement and Related Products.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions of Sections 4(*b*) and 14(*c*), issued Sept. 9, 1958, of the Standard Specifications and Methods of Test for Asbestos-Cement Siding Shingles and Clapboards (**C 223 – 55**)[2] be approved for reference to letter ballot of the Society for adoption as standard.[3]

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 5.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications and Methods of Test for Flat Asbestos-Cement Sheets (C 220 – 55):[2]

Title.—Delete the words "and Methods of Test."

Section 1.—Delete from the first line the words "and methods of test."

Section 3.—In Paragraph (*a*) add the words "not less than" after the words "shall be," and delete the word "minimum." In Paragraph (*b*) delete the word "minimum."

Table I.—Delete the words "Minimum" and "Requirements" from the second column heading.

Table II.—Delete the word "minimum" from the second column heading.

Section 4.—Delete the second and third sentences.

Table III.—Add the word "Nominal" to the table title. Delete the word "nominal" from the column headings.

Table IV.—Add additional thicknesses as shown in the accompanying Table I.

Sections 10 to 14.—Delete these sections on Methods of Testing.

Standard Specifications and Methods of Test for Asbestos-Cement Roofing Shingles (C 222 – 55):[2]

Title.—Delete the words "and Methods of Test."

Section 1.—In Paragraph (*a*) delete the words "and methods of test."

In Paragraph (*b*), under *American Method*, replace the second sentence with the following: "They are intended to be laid with a minimum (2 in.) head lap and with no side lap and to provide double coverage. They may be made either as individual or multiple units to simulate the appearance of individual shingles."

TABLE I.—ADDITIONS TO TABLE IV, SPECIFICATIONS C 220.

Nominal Thickness, in.	Average Thickness, in.	
	Minimum	Maximum
½(0.500).........	0.470	0.530
⅝(0.625).........	0.595	0.655

Add a new paragraph on *Strip Shingles*, to read as follows:

> *Strip Shingles.*—Uniform thickness, various shapes and designs to provide top lap only and shingle coverage under the butt joints, with the design such that it provides only single coverage.

Replace the paragraph on *Dutch or Scotch Method* with the following:

> *Dutch or Scotch Method* (*including Ranch design*).—Uniform thickness with straight or irregular edges and designed to be laid with a lap at the top and on one side of each shingle.
>
> *Dutch*—square in shape.
>
> *Ranch*—oblong-rectangular, with the greater dimension horizontal.

Delete the paragraphs on *American Method Strip* and *Ranch Type*.

Section 3(*a*).—In Paragraphs (*a*) and (*b*) delete the word "minimum."

Table I.—Revise as shown in the accompanying Table II.

---

[2] 1958 Book of ASTM Standards, Part 5.

[3] Through an inadvertence, this recommendation, although approved by letter ballot in the committee, was not included in the report of the committee as preprinted. It accordingly was not included on the regular Society letter ballot but was covered separately through a note in the ASTM BULLETIN.—Ed.

Section 6(d).—Revise to read as follows:

> (*d*) *Holes for Nails and Fasteners.*—Holes for nails and fasteners shall be provided in the units during manufacture and shall be so placed as to provide at least the minimum lap as specified in Table I and to allow for proper application of necessary clips or storm fasteners.

TABLE II.—REVISION OF TABLE I, SPECIFICATIONS C 222.

Type	Width (Horizontal), in.	Height (Vertical), in.	Minimum Lap, in.		
			Top[a, b]	Head[a, b]	Side[a, b]
American Method	8 to 32	12 to 18½	...	2	...
Strip Shingle	12 to 32	variable	2	...	...
Dutch or Scotch	16	16	3	...	4
Ranch	24	12	2½	...	4
French or Hexagonal	16	16	3	...	3

[a] Headlap with shingles that provide double coverage and top lap with shingles that provide single coverage is defined as the shortest distance between the lower edge of a course of shingles and the most proximate underlying area of roof deck not covered by the preceding courses of shingles. At butted side joints, between individual units, complete and continuous coverage shall be provided by the undercourse shingles. Side lap has a similar meaning in respect to the shortest horizontal distance from the exposed side edge to uncovered roof deck area.

[b] The unit dimensions and laps given in this table are an indication of customary current manufacture and shall not be construed to limit or fix the width, length, or lap dimensions of shingles that conform to all other requirements of this specification.

Section 8.—Change the second sentence of Paragraph (*b*) to read: "The nails shall be of such length as to hold securely in the deck." In Paragraph (*d*), in the second line of the first sentence replace "from" with "of." In the second sentence delete "made of."

Sections 13 to 17.—Delete these sections on Methods of Testing.

Standard Specifications and Methods of Test for Asbestos-Cement Siding Shingles and Clapboards (C 223 – 55):[2]

Title.—Delete the words "and Methods of Test."

Section 1.—Change the first sentence of Paragraph (*a*) to read: "(*a*) These specifications cover asbestos-cement siding including accessories, designed to provide the weather exposed sidewall surfaces of buildings."

Add a paragraph on Sheets to read as follows: "*Sheets.*—Uniform thickness, rectangular in shape, larger in unit sizes than shingles or clapboards.

TABLE III.—REVISION OF TABLE I, SPECIFICATIONS C 223.

Type	Width, in.	Length, in.	Minimum Top Lap,[a] in.
Siding Shingles	8 to 16	24 to 48	1
Clapboards	8 to 16	48 to 96	1
Sheets[b]	16 to 48	48 to 144	1 (unless flashed)

[a] Top lap is defined as the shortest distance between the lower edge of a course of siding shingles or clapboards and the most proximate area of side wall not covered by the preceding course.

[b] Flat sheets are also covered by ASTM Specifications C 220.

Change Paragraph (*b*) to read as follows: "(*b*) All measurements and tests necessary for determining the conformity of the asbestos-cement siding with these specifications shall be made in accordance with the methods covered in Sections 12 to 16."

Section 2.—In the first line delete the words "shingles and clapboards."

Section 3(a).—Delete the word "minimum."

Section 4(a).—In the first line replace the words "Shingles and clapboards" with "Asbestos-cement siding."

Table I.—Revise as shown in the accompanying Table III.

Section 5(a).—In line 10 replace the words "shingles or clapboards" with the word "siding."

Section 6(a).—Replace the words "shingles or clapboards" with the word "siding."

Section 7(a).—In line 3 replace the word "punched" with the word "provided." In line 5 delete the words "or clapboard."

Section 8(a).—Change the words "shingles or clapboards" to read "siding."

Section 9(a).—Change the words "shingles or clapboards" to read "siding."

Sections 12 to 16.—Delete these sections on Methods of Testing.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Methods of Test (S. E. Williams, chairman).—During the year the Method of Test for Organic Fiber Content of Asbestos-Cement Products was completed and is recommended to the Society as appended to this report.[1] Methods of tests for all products except corrugated sheets as presently contained in the specifications were separated and combined into two separate tentatives as appended hereto.[1] A method of test for uncombined calcium hydroxide to replace the alkalinity test is being considered.

Subcommittee II on Specifications (M. V. Engelbach, chairman) completed the revisions of Specifications C 296 C 428. Revisions of Specifications C 220, C 222, and C 223.

Subcommittee III on Research (William Lerch, chairman) is preparing a proposed method of test for determination of organic fiber in asbestos-cement products. Cooperation with Subcommittee I was also given in a proposed method of test for uncombined calcium hydroxide.

Subcommittee IV on Nomenclature (W. A. Heausler, chairman) prepared the Tentative Definitions of Terms Relating to Asbestos-Cement and Related Products, noted elsewhere in this report.

This report has been submitted to letter ballot of the committee, which consists of 33 voting members; 22 members returned their ballots, of whom 21 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. V. FRIEDLAENDER,
Chairman.

SIMON COLLIER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-17 presented to the Society through the Administrative Committee on Standards tentative revisions of Standard Specifications for Flat Asbestos-Cement Sheets (C 220 – 55). The tentative revisions were accepted by the Standards Committee on September 29, 1960, and they appear in the 1960 Supplement to Book of ASTM Standards, Part 5.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-18

ON

NATURAL BUILDING STONES*

Committee C-18 on Natural Building Stones held one meeting during the year: on March 25, 1960, at Washington, D. C. Meetings of the subcommittees, including several of the working groups for specifications, were held concurrently with the general meeting.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. W. Key.
Vice-Chairman, Theodore I. Coe.
Secretary, F. S. Eaton.

The committee consists of 20 members, of whom 8 are classified as producers, 6 as consumers, and 6 as general interest members. There are two consulting members.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specification for Structural Granite (**C 422 – 58 T**).[1]

SUBCOMMITTEE ACTIVITIES

Subcommittee II on Nomenclature and Definitions (L. W. Currier, chairman) is considering the need for a better classification in cut stone, ashlar, rubble, as well as certain textural terms.

Subcommittee IV on Specifications (F. S. Eaton, chairman).—The Task Group on Specifications for Limestone is reviewing an initial draft of a proposed specification, particularly in connection with strength requirements. The task group is being reorganized for this purpose.

The Task Group on Specifications for Sandstone is being reactivated for the purpose of reviewing an initial draft of a proposed specification for sandstone.

The Task Group on Specifications for Granite is considering objections to Tentative Specification C 422 which may lead to moderate revisions. Additional test criteria will be reviewed as a basis for evaluation, including sonic testing. The preparation of a standard sample for testing purposes is a problem which the task group will consider.

The Task Group on Specifications for Marble has reviewed the latest draft of a proposed specification. Action was taken at the meeting of Committee C-18 to submit this proposed specification to letter ballot of the committee, with certain editorial changes agreed upon.

Respectfully submitted on behalf of the committee,

L. W. CURRIER,
Chairman.

F. S. EATON,
Secretary.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-20
ON
ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held two meetings during the year: at ASTM Headquarters in Philadelphia, Pa., on Oct. 12 and 13, 1959, and at the U. S. Forest Products Laboratory, Madison, Wis., on April 14 and 15, 1960.

At the meeting of the committee on April 15, 1960, action was taken to recommend to Committee E-5 that the proposed Method of Test for Measuring Surface Flammability of Building Materials Using an 8 ft Tunnel Furnace be promulgated as a tentative.

The committee consists of 72 individuals representing 22 producers, 11 consumers, and 31 general interest members. There are 62 total votes.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. N. Hamme.
Vice-Chairman, H. J. Sabine.
Secretary, Ralph Huntley.

Revision of Tentative

The committee recommends revisions as follows of the Tentative Method of Test for Sound Absorption of Acoustical Materials in Reverberation Rooms (**C 423 - 58 T**)[1] and continuation of the method as tentative:

Section 3(d).—Starting with the second sentence, change to read as follows:

> Also, since the absorption of the air in the reverberation room varies with humidity and temperature these two variables should be kept constant during each pair of measurements. It is suggested that the relative humidity be maintained above 50 per cent. A practical method is to make frequent measurements of the absorption of the empty room. If the humidity is not controlled, allowance may be made in the computations in the following way:
>
> (remainder of paragraph unchanged)

Section 4(e).—Delete this entire paragraph.

Section 7(d).—Change to read as follows:

> (*d*) *Number of Measurements*.—Repeated measurements of the decay rate under the same conditions will be expected to show some scatter. At each frequency the average of a group of measurements shall be used and the size of the group shall be determined by the method outlined in Section 4 of Recommended Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process (ASTM Designation: E 122).[6] The maximum allowable sampling error shall be 1 per cent.

---

[*] Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 5.

[6] The formula suggested is:

$$n = \left(\frac{2.5v'}{e}\right)^2$$

where:

n = size of the group of measurements,
e = the allowable sampling error in per cent,
v' = the coefficient of variation in per cent determined in advance for each frequency,
$v' = \dfrac{\sigma' 100}{\bar{X}'}$,
σ' = standard deviation of a large number of measurements,
$\sigma' = \sqrt{\dfrac{\Sigma (X_i - \bar{X}')^2}{n'}}$ or $\sqrt{\dfrac{\Sigma X_i^2}{n'} - \bar{X}'^2}$,
X_i = an individual measurement,
$\bar{X}'$ = the average of a large group of preliminary measurements,
n' = the size of the large group of preliminary measurements, and

2.5 = a constant chosen to make the degree of certainty 98.75 per cent that the average of a small number of measurements, n, will differ from the average of the large group by less than 1 per cent.

Example.—A large number of measurements are made of the decay time at a certain frequency (say 1000 measurements at 500 cps) and an average value is obtained of $T = 3.00$ sec with $\sigma' = 0.09$. The question is how many measurements should be made to obtain a satisfactory average. One thousand are probably more than are needed.

The coefficient of variation is given by:

$$v' = \frac{0.09 \times 100}{3} = 3\%$$

Substituting in the formula and putting e = 1 per cent:

$$n = (2.5 \times 3)^2 = 56.25$$

The conclusion is that a satisfactory average value of T will be obtained if a group of 60 measurements are made.

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sound Absorption and Attenuation (H. J. Sabine, chairman) is recommending the revision of Method C 423 listed earlier in this report. Task groups have been working on three methods of test: the box method, the horn coupler, and the close-up method. Correlation between the results of measurements by the acoustic tube method and the reverberation room method is being made, and the first draft of the method of test for sound attenuation by ceilings is in preparation.

Subcommittee II on Flame Resistance (Wallace Waterfall, chairman) has been in liaison with Committee E-5 on Fire Tests of Materials and Construction on the several methods of test for flame resistance.

Subcommittee III on Maintenance (W. M. Rees, chairman) is preparing a method of test for repaintability of acoustical materials.

Subcommittee IV on Application (R. Lindahl, chairman).—The Task Force on Mechanical Suspension Systems is preparing test methods and is working with other interested groups in an attempt to write a performance specification for mechanical suspension systems for acoustical products. Liaison has also been made with Committee E-6 on Methods of Testing Building Constructions.

Subcommittee V on Basic Physical Properties (W. A. Jack, chairman) is preparing a proposed tentative method of test for light reflectance of acoustical materials by the integrating sphere reflectometer for subcommittee letter ballot.

A proposed method of test for measuring the air-flow resistance of acoustical materials based on "An Apparatus for Measuring Air-Flow Resistance of Acoustical Materials" by H. J. Sabine (ASTM BULLETIN, January 1956) is being studied by the subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 62 voting members; 43 members returned their ballots, all of whom voted affirmatively.

Respectfully submitted on behalf of the committee,

R. N. HAMME,
Chairman.

RALPH HUNTLEY,
Secretary.

---

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-21

ON

CERAMIC WHITEWARES AND RELATED PRODUCTS*

Committee C-21 on Ceramic Whitewares and Related Products held two meetings during the year: in Bedford Springs, Pa., on Oct. 1, 1959, and in Philadelphia, Pa., on April 25, 1960.

The committee consists of 80 voting members of whom 46 are classified as company, 27 as individual, 4 as government, and 3 as ASTM memberships. (The votes are classified as 42 producers, 11 consumers, and 27 general interest members.) Because of multiple representation for several company and government memberships there are 118 persons with current voice in voting.

The officers elected for the ensuing term of two years are as follows:

Chairman, Neil T. Morrison.

Vice-Chairman, William C. Mohr.

Secretary, Richard C. Phoenix.

Members-at-Large, R. W. Cline and W. W. Perkins.

A By-laws Task Group is in the process of revising the committee by-laws to bring them into conformance with the reorganization effected in 1958.

The work initiated in Committee C-21 by Subcommittee VII on Nonmetallic Magnetic Materials and by Task Group D on Titanates and Zirconates has been transferred to the newly formed Committee C-25 on Ceramics for Electronics.

Task Group B on Cermets, having fulfilled its survey mission, was discharged.

Revisions of Tentative

The committee recommends revisions as follows of the Tentative Definitions of Terms Relating to Ceramic Whitewares and Related Products (**C 242 - 58 T**)[1] and continuation of the definitions as tentative:

Revised Definitions.—Revise the following definitions to read as shown:

Ball Milling.—A method of grinding and mixing material with or without liquid, in a rotating cylinder or conical mill partially filled with grinding media such as balls or pebbles.

Casting, Solid.—Forming ceramic ware by introducing a body slip into a porous mold which usually consists of two major sections, one section forming the contour of the outside and the other forming the contour of the inside of the ware and allowing a solid cast to form between the two mold faces.

Clay.—A natural mineral aggregate, consisting essentially of hydrous aluminum silicates; it is plastic when sufficiently wetted, rigid when dried en masse, and vitrified when fired to a sufficiently high temperature.

Drying.—Removal by evaporation, of uncombined water or other volatile substance from a ceramic raw material or product, usually expedited by low-temperature heating.

Embossed.—Decorated in relief on the surface of the ware.

Faience Ware.—Formerly a decorated earthenware with an opaque glaze, but currently designating a decorated earthenware having a transparent glaze.

Glazed Tile, Extra Duty Glaze.—Tile with a durable glaze that is suitable for light duty floors and all other surfaces on interiors where there is no excessive abrasion or impact.

Majolica.—Formerly an earthenware with an opaque luster glaze and overglaze colored decorations, but currently designating any decorated earthenware having an opaque glaze.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 5.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Crazing Resistance of Fired Glazed Whitewares by Autoclave Treatment (**C 424 – 58 T**)[1] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Definitions of Terms Relating to Ceramic Whitewares and Related Products (**C 242 – 56**),[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Revised Definitions.—Revise the following definitions to read as shown:

Absorption.—The relationship of the weight of the water absorbed by a ceramic specimen, subjected to prescribed immersion procedure, to the weight of the dry specimen, expressed in per cent.

Bone China.—A translucent china made from a ceramic whiteware body composition containing a minimum of 25 per cent bone ash.

Bright Glaze.—A colorless or colored ceramic glaze having high gloss.

Clear Glaze.—A colorless or colored transparent ceramic glaze.

Mat Glaze.—A colorless or colored ceramic glaze having low gloss.

Primary Clay (Residual Clay).—A clay which remains geologically at its site of formation.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (A. S. Watts, chairman) has been engaged in a critical study of all terms and definitions published in Standard Definitions C 242 – 56. A number of proposed changes were circulated in the subcommittee, resulting in acceptance of 18 changes which are now being circulated in Committee C-21. Additional suggested revised definitions for Standard Definitions C 242 – 56 are being developed for consideration of the subcommittee. The subcommittee also approved six proposed changes in Tentative Definitions C 242 – 58 T.

Subcommittee VI on Tile submitted a list of 15 proposed tile definitions which are now being circulated in Committee C-21.

Subcommittee II, Editorial (R. F. Geller, chairman) was reorganized during the year, having lost through death Edward Schramm, and has under active study all test methods and specifications promulgated by Committee C-21.

Subcommittee III on Fundamental Properties (W. C. Mohr, chairman) has completed its study on the effect of detergents on overglaze decoration and has discharged the responsible task group. Tests for lead solubility of glazes are under consideration; this involves an investigative program to determine proper reagents and specimens. Through its chairman, this subcommittee sponsored a session on processing controls at the Fall Meeting of the Whitewares Division of the American Ceramic Society at Bedford Springs, Pa., in October, 1959. A session on forming methods was sponsored at the Spring Meeting of the Whitewares Division of the American Ceramic Society at Philadelphia, Pa., in April, 1960.

Subcommittee IV on Clays (G. W. Phelps, chairman) continued its work on a proposed particle size technique, a method for determination of organic matter in clays, and a method for determination of soluble sulfate in clays.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Nuclear Applications (J. H. Handwerk, chairman) is directing its efforts to formation of special groups designed to deal with the need for standards in the area of nuclear applications. In this connection a Task Group on Nuclear Grade Graphite has been formed under the chairmanship of Mr. W. C. Riley. Subcommittee V organized and presented a one-day symposium in the area of nuclear applications at the ASTM Third Pacific Area National Meeting at San Francisco in October, 1959.[3] The uses of ceramic materials as moderators, fuels, control materials, and structural components were discussed. Materials touched upon included beryllium oxide, uranium oxide, graphite, silicon carbide, and refractory uranium compounds such as carbides and sulfides.

Subcommittee VI on Ceramic Tile (Arno M. Illing, chairman) held meetings in May and October, 1959 during the regular meetings of Committee C-21, and at Princeton, N. J., on Jan. 16, 1960. Four proposed test methods are in the hands of the Editorial Subcommittee for editing, and a proposed test method for electrical resistance of conductive ceramic tile has been approved and submitted to Committee D-9 on Electrical Insulating Materials for review. Fifteen definitions for ceramic tile terms have been submitted to the committee for ballot.

Subcommittee VII on Nonmetallic Magnetic Materials (H. H. Kedesdy, chairman) was advanced from Task Group status in May, 1959. Task groups on microwave ferrites (M. Eisenberg, chairman), computer application (E. T. Wierman, chairman), and pulse transformer work (C. W. Lundberg, chairman) were formed. In December, 1959, the activities of Subcommittee VII were transferred to Committee C-25 on Ceramics for Electronics.

Task Group C on Nonplastics (W. P. Shulhof, chairman) was reactivated during the year for the expressed purpose of setting up particle-size procedures for nonplastic powders.

The major activity of Task Group C has been the building up of an active membership. A suitable core of members has been found: William P. Shulhof, J. C. Griffiths, Ronald H. Lester, G. W. Phelps, and D. W. Utley. Each member has been requested to review his experience in the particle size analysis field, give the specific objectives he thinks the group should have, advise as to the equipment available, and compile a bibliography on size analysis in his own area. From this information, an agenda will be constructed for the first meeting of the group tentatively scheduled for June 29 during the ASTM Annual Meeting in Atlantic City. Other individuals have been approached to aid the group as consultants.

Task Group F on Foamed Ceramics (A. J. Metzger, chairman *pro tempore*) is in the process of enlisting membership and establishing a list of test methods for evaluation of foamed ceramics.

Task Group G on Nuclear Grade Graphite (W. C. Riley, chairman) held an organizational meeting on Feb. 10 and 11, 1960, at the Franklin Institute Laboratories in Philadelphia. A 14-member group has been formed, representing a wide area of interests. A tentative list of specifications is being drawn up and physical and chemical data for various commercially available graphites are being assembled. A meeting is planned for the Annual Meeting of the American Nuclear Society, June 12 to 15, 1960, in Chicago.

---

[3] The papers presented at this symposium are included in *ASTM STP No. 276.*

This report has been submitted to letter ballot of the committee, which consists of 80 voting members; 61 members returned their ballots, of whom 57 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

M. D. BURDICK,
Chairman.

G. W. PHELPS,
Secretary.

REPORT OF COMMITTEE C-22
ON
PORCELAIN ENAMEL*

Committee C-22 on Porcelain Enamel held two meetings during the year: on Sept. 29 and 30, 1959, in Columbus, Ohio, as guests of Battelle Memorial Institute; and on Feb. 3 and 4, 1960, in Chicago, Ill., during ASTM Committee Week.

The committee consists of 51 members of whom 44 are voting members; 25 are classified as producers, 5 as consumers, and 14 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. N. Harrison.
First Vice-Chairman, D. G. Bennett.
Second Vice-Chairman, E. E. Howe.
Secretary, G. H. Spencer-Strong.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Fusion Flow of Porcelain Enamel Frits (Flow-Button Method) (**C 374 - 55 T**),[1] and
Torsion Resistance of Laboratory Specimens of Porcelain Enameled Iron and Steel (**C 409 - 57 T**).[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Definitions of:

Terms Relating to Porcelain Enamel (**C 286 - 58 T**).

Tentative Methods of Test for:

Impact Resistance of Porcelain-Enameled Utensils (**C 284 - 51 T**), and
Warpage of Porcelain-Enameled Flatware (**C 314 - 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (B. J. Sweo, chairman) has completed its work on the evaluation of porcelain enamel and ceramic coatings on high- and low-alloy steels at elevated temperatures and its survey of engineering data relating to enamel metal system. It has begun work on three new projects: (1) ceramic adhesives, (2) gases in porcelain enamels, and (3) fracture defects such as hairlining and tearing.

Subcommittee II on Nomenclature (E. E. Howe, chairman) has continued its work on definitions of terms used in the porcelain enamel industry.

Subcommittee III on Education (L. S. O'Bannon, chairman) has continued its contacts with the technical press and is largely responsible for six new members added to the committee during the year.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 5.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Materials (J. K. Magor, chairman) has eight active projects under way and is responsible for the recommendations concerning Methods C 374 and C 409 noted earlier in this report.

Subcommittee V on Finished Products (J. C. Richmond, chairman) has 10 active projects under way, two of which (the proposed methods of test for continuity of coating and test for spalling resistance of porcelain enamel on aluminum) should evolve into proposed tentative methods of test within the year.

This report has been submitted to letter ballot of the committee, which consists of 44 voting members; 35 members returned their letter ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

REPORT OF COMMITTEE C-23
ON
SORPTIVE MINERAL MATERIALS

The Board of Directors at its meeting of May 8, 1956, authorized the formation of a new technical committee, Committee C-23 on Sorptive Mineral Materials. The organization meeting of Committee C-23 was held on Feb. 5, 1957, in Philadelphia, Pa. Meetings were held on June 20, 1957, in Atlantic City, N. J., Sept. 20, 1957, and Oct. 15, 1958, in Detroit, Mich., and Oct. 23, 1959, in New York, N. Y. Subcommittee meetings were held on the same dates and in addition, on April 10, 1958, in Detroit, Mich.

The committee consists of 23 members, of whom 13 are classified as producers, 9 as consumers, and 1 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, P. R. Izdepski.
Vice-Chairman, L. M. Mank.
Secretary, R. L. Shirley.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definitions and Nomenclature (H. M. Gwyn, Jr., chairman) has prepared a list of definitions for terms used in the field of sorptive mineral materials. This list is being reviewed by the committee for additions and corrections.

Subcommittee II on Sampling and Testing (M. G. Kramer, chairman) has prepared proposed tentative methods for loss on ignition, free moisture, sampling, and sieve analysis. These methods will be submitted to the Society upon completion of the main committee ballot.

Task Group A on Contact with Industry (L. M. Mank, chairman) has gathered information on test methods used by the industry for evaluation of floor absorbents. Contacts with more than 50 large consumers of sorptive mineral materials have been made.

Task Group B on Review of the Literature (N. W. Berst, chairman) has completed a bibliography on test methods for sorptive mineral materials used for floor surface maintenance and safety, containing 147 references. This bibliography will serve as a guide for the methods developed by the committee.

Task Group C on Development of Test Methods (J. E. Fennell, chairman) has completed interlaboratory studies on methods for bulk and tamped density and solubility in water. Further work on these methods will be made. Methods for dust content and for water and mechanical breakdown are being cooperatively tested. Methods for absorbency, slipperiness, abrasiveness, fire resistance, and ease of removal are being developed.

Subcommittee III on Performance Standards (Samuel Jaffee, chairman) is reviewing existing simulated service tests for efficiency of floor absorbents. The subcommittee is reviewing plant evaluation tests used by various organizations in order to determine what are considered the most significant properties and how they were established.

This report has been submitted to letter ballot of the committee, which consists of 23 members; 18 members returned their ballots, of whom 17 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. R. BALDEN,
Chairman.

R. L. SHIRLEY,
Secretary.

REPORT OF COMMITTEE C-24
ON
JOINT SEALANTS*

The Board of Directors of ASTM at their May 1959 meeting authorized the formation of a technical committee to study building sealants. A conference of individuals representing various phases of the sealants industry met at ASTM Headquarters on June 10, 1959. A Steering Committee was formed which consisted of the following:

C. D. Barlet, Armstrong Cork.
R. K. Humke, Minnesota Mining and Manufacturing Co.
H. Kelfer, Sterling Quality Products (ASTM Committee D-1).
W. Koppes, Architectural Consultant.
T. P. Martin, Pittsburgh Plate Glass Co.
G. J. Schulte, Minnesota Mining and Manufacturing Co.
J. R. Panek, Thiokol Chemical Corp.
D. E. Parsons, National Bureau of Standards (Chairman).

The Steering Committee met on Sept. 1, 1959 at ASTM Headquarters, at which time they formulated a tentative invitation list as well as organized plans for the organizational meeting.

The organization meeting of Committee C-24 on Joint Sealants was held on Sept. 15, 1959, at ASTM Headquarters in Philadelphia, Pa., with 67 members and guests in attendance. The initial membership list consisted of 21 producers, 26 consumers, and 13 general interests.

The scope of the committee is as follows:

> "The promotion of knowledge, stimulation of research, and the development of methods of tests, specifications, and nomenclature of materials for sealing joints and openings. Such sealants include caulking and glazing compounds and preformed shapes. These activities will be coordinated with those of other relevant committees of ASTM."

The following officers were elected:

Chairman, W. Koppes.
Vice-Chairman, R. K. Humke.
Secretary, J. R. Panek.
Membership Secretary, H. Kleinhans.

Three Subcommittees were formed:

Subcommittee I on Nomenclature,
Subcommittee II on Bulk Compounds, and
Subcommittee III on Preformed Shapes.

Pending the formulation of by-laws for the committee, the group authorized the officers, the Steering Committee, and two members-at-large to serve as the Temporary Executive Committee. The two members-at-large were: J. LaFleur, New York State Thruway, and Mr. G. L. McGill, The Martin Co.

A By-laws Subcommittee, consisting of T. P. Martin and J. R. Panek, was appointed and a set of by-laws was drawn up and presented for review to the Temporary Executive Committee, which met on Nov. 12, 1959, at ASTM Headquarters. The by-laws were approved by letter ballot sent out on Nov. 23, 1959. With this move, the Temporary Executive Committee was replaced by the official Executive Committee, which consists of the four officers, the chairmen of the three subcommittees and the two members-at-large, G. L. McGill and H. H. Morgan, appointed by the chairman.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (G. F. Lamb, chairman) held a meeting on May 6, 1960, at Washington, D. C. to discuss nomenclature of terms used in Committee C-24 activities.

Subcommittee II on Bulk Compounds (F. Frybergh, chairman; J. Amstock, secretary) held meetings on Jan. 21 at Philadelphia, Pa., and on Feb. 26, 1960 at New York City.

Three task groups have been set up and their activities are as follows:

Task Group I on Methods of Tests (B. Pozniak, chairman).

Subgroup A on Tests on Adhesion in Tension and Accelerated Aging (B. Pozniak, chairman).

Subgroup B on Tests on Flow, Sag, and Application Life (R. Humke, chairman).

Subgroup C on Tests on Staining (A. Hochman, chairman).

Task Group II on Specifications (F. Frybergh, chairman).

Task Group II has taken the proposed American Standard Specification for Sealants for the Building Trade and revised the format and outline.

Task Group I and its subgroups will be responsible for deriving the tests and setting up "round-robins."

Subcommittee III on Preformed Shapes (R. McKinley, chairman; M. F. Torrence, secretary) held a meeting on Feb. 24, 1960, at ASTM Headquarters. Two task groups have been formed, namely:

Subgroup A on Architecture and Construction (C. B. Monk, Jr., chairman).

Subgroup B on Underground Pipe (F. P. Valenziano, chairman).

It is agreed that the temporary scope of the subcommittee would embrace application needs of architecture and constructions including mobile homes; of concrete pipe and paving; of automobile, aircraft and vehicular frames, windows and doors.

Respectfully submitted on behalf of the committee,

W. KOPPES,
Chairman.

J. R. PANEK,
Secretary.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 22 to 24, 1959, in Atlantic City, N. J., in connection with the Annual Meeting of the Society, and in Washington, D. C., on February 15 to 17, 1960.

At the June, 1959, meeting, W. G. Vannoy, Pigments Department, E. I. du Pont de Nemours & Co., Inc., Chestnut Run, Del., presented a paper on "House Paint Trends." The paper showed the importance of proper pigment selection and the physical properties imparted by several classes of white pigments in the various types of house paints. New types of house paints such as the alkyd blister-resistant and stain-resistant types were discussed as well as the use on new wood of alkyd primers with emulsion paint top coats.

At the February, 1960, meeting, C. A. Lucchesi, Analytical Research Department, Sherwin-Williams Co., Chicago, Ill., presented a paper on "Analytical Spectroscopy in the Coatings Industry." The paper discussed the theory and applications as related to paint chemistry of four fields of spectroscopy: ultraviolet absorption, infrared absorption, X-ray fluorescence, and flame photometry.

Committee D-1 has continued its close cooperation with the Federation of Societies for Paint Technology. Ninety-six standards have been jointly approved by the two organizations. The Joint Federation-ASTM Committee on Paint, Varnish and Lacquer (J. C. Weaver, chairman) serves as the permanent liaison group between the two organizations. In the past year it has cooperated with the Federations' Standards and Methods of Test Committee in indexing all ASTM methods of test and specifications relating to paints as well as methods of test published by the Federal Government and the Canadian Government.[1]

The Committee on Intercommittee Relations (E. W. Fasig, chairman) is composed of the official representatives of Committee D-1 on other ASTM committees. The purpose of the committee is to maintain close contact with the work of those committees whose activities are of direct interest to Committee D-1, and also to represent the interests of Committee D-1 on such other committees. In the past year, upon the recommendation of Subcommittee IV on Traffic Paint, representatives have been appointed to Committee D-4 on Road and Paving Materials. In addition, and also upon the recommendation of Subcommittee IV, arrangements are being made for official Society representation on the Highway Research Board and the Institute of Traffic Engineers.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] See Comprehensive Index of Methods of Test for Paints and Paint Materials, *Official Digest*, Federation of Societies for Paint Technology, August, 1960, Part 2. Copies at $1.00 each are available from the Federation and from ASTM Headquarters.

The Advisory Committee established the office of Assistant Secretary of Committee D-1 and elected C. A. Lominska to fill the new post.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. T. Pearce.
Vice-Chairman, J. C. Weaver.
Secretary, C. A. Lominska.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications, methods, and recommended practices as appended hereto:[2]

Tentative Specifications for:

- Isobutyl Alcohol (Isobutanol), and
- Isobutyl Acetate (93 per cent Minimum Ester Grade).

Tentative Methods of:

- Test for Qualitative Determination of Fish Oil in Drying Oils and Drying Oil Fatty Acids,
- Test for Water Miscibility of Isopropyl Alcohol (Isopropanol),
- Test for Water Solubility of Acetone,
- Test for Permanganate Time of Tricresyl Phosphate,
- Test for Dilution Ratio in Cellulose Nitrate Solutions for Active Solvents, Hydrocarbon Diluents, and Cellulose Nitrates,
- Test for Hiding Power of Nonchromatic Paints,
- Test for Phthalate Ester Color of High-Gravity Glycerin (An Historical Review and Supporting Data on this method appears in Appendix I[3]),
- Test for Viscosity of Resin Solutions, (The results of an interlaboratory study on Viscosity of Resin Solutions are given in Appendix III.[3a])
- Test for Urea Content of Nitrogen Resins, (Experimental data on Urea Content of Nitrogen Resins are given in Appendix II.[3b])
- Test for Hydrolyzable Chlorine Content of Liquid Epoxy Resins,
- Test for Efflorescence of Interior Latex Paints,
- Test for Elongation of Attached Coatings of Paint, Varnish, Lacquer, and Related Products with Cylindrical Mandrel Apparatus,
- Preparation of Aluminum Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products,
- Making and Preparing Concrete and Masonry Panels for Testing Paint Finishes,
- Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting (as published in ASTM *Proceedings*, Vol. 56, 245 (1956),
- Preparation of Magnesium Alloy Surfaces for Painting (as published in ASTM *Proceedings*, Vol. 56, p. 249 (1956),
- Water Fog Testing of Organic Coatings, and
- Preparation of Hot-Dip Aluminum Surfaces for Painting.

Tentative Recommended Practice for:

- Visual Evaluation of Color Differences of Opaque Materials.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Color Difference Using the Hunter Color Difference Meter (D 1365 – 55 T):[4]

Title.—Change to read: "Tentative Method of Test for Color Difference Using Color Difference Meters of the Hunterlab or Gardner Types."

Section 4(a).—Change the first sentence to read: "The Hunterlab or Gardner Color Difference Meters shall be used (see Note.)"

New Note.—Add a note following Section 4(*a*) to read as follows:

NOTE.—Two types of color difference meters were developed by R. S. Hunter. One is a barrier-layer cell type supplied by the Gardner Laboratory, Inc., 5521 Landy Lane, Washington 16, D. C. The other is a vacuum tube type supplied by Hunter Associates Laboratory, 5421 Brier Ridge Rd., McLean, Va.

Section 7(a)(1).—Delete reference (8).

Section 8(c).—Change to read: "Identify the instrument used by maker's name and model number. The standards used shall be identified."

---

[2] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 8.

[3] See p. 311.

[3a] See p. 315.

[3b] See p. 314.

[4] 1958 Book of ASTM Standards, Part 8.

Section 9(a).—Change to read as follows:

9. (*a*) Limited tests conducted to determine instrument repeatability and reproducibility for color difference magnitudes of less than 10 units revealed the following:

	Color Difference, ΔE	Lightness and Chromaticity Difference, ΔL, Δa, Δb
Repeatability........	0.2	0.1
Reproducibility......	0.3	0.2

NOTE.—Repeatability is defined as the standard deviation of results obtained by one operator using one instrument in one laboratory.

Reproducibility is defined as the standard deviation of results obtained by different operators using the same type instrument in different laboratories.

References.—Delete reference number 8.

Tentative Method of Test for Color Difference Using the Color Eye (D 1495 – 57 T):[4]

Section 4.—Change the value of *b* to read as follows:

$$b = 40\,(0.4(V_y - V_z)) = 16\,(V_y - V_z)$$

Section 8.— Add the heading: "8. (*a*) *Routine Comparison Method*:" and designate the present Paragraphs (*a*) to (*f*) as Items (*1*) to (*6*). Add a new Paragraph (*b*) to read as follows:

(*b*) *Substitution Method for Highest Precision:*

(*1*) Same as previous Paragraph 8 (*a*).

(*2*) Place white vitrolite block or other suitable standard color (which may be similar to the reference specimen) in the "standard" position and the reference specimen in the "sample" position. Set the filter selector to *X* and with duodial, set meter to 100.

(*3*) Remove the reference specimen and substitute the test specimen in the sample position and retain *percentage deviation* for *X* to the nearest 0.1 per cent. Make three readings on representative portions of the test specimen, unless instructed otherwise. Use high sensitivity, except where low sensitivity is necessary to bring meter readings on scale.

(*4*) Repeat steps (*2*) and (*3*) with filter selector *Y* and *Z* positions in turn.

Tentative Method of Test for Color Change of White Architectural Enamels (D 1543 – 58 T):[5] Revise as appended hereto.[6]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends that the Standard Method of Test for Aniline Point and Mixed Aniline Point of Hydrocarbon Solvents (**D 1012 – 51**)[4] be revised as appended hereto,[6] and reverted to tentative.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Distilled Soybean Fatty Acids (**D 1537 – 58 T**),
Distilled Linseed Fatty Acids (**D 1538 – 58 T**), and
Dehydrated Castor Acids (**D 1539 – 58 T**).

Tentative Methods of Test for:

Mass Color and Tinting Strength of Color Pigments (**D 387 – 52 T**),
Temperature-Change Resistance of Clear Nitrocellulose Lacquer Films Applied to Wood (**D 1211 – 57 T**),
Unsaponifiable Matter in Alkyd Resins and Resin Solutions (**D 1397 – 58 T**),
Volatile Matter in Tricresyl Phosphate (**D 1468 – 57 T**),
Density of Paint, Varnish, Lacquer, and Related Products (**D 1475 – 57 T**),
Heptane Miscibility of Lacquer Solvents (**D 1476 – 57 T**),
Total Iodine Value of Drying Oils and Their Derivatives (**D 1541 – 58 T**),
Viscosity of Transparent Liquids by Bubble Time Method (**D 1545 – 58 T**),
Melamine Content of Nitrogen Resins (**D 1597 – 58 T**),

---

[5] Accepted as Tentative in June, 1958, but temporarily withheld from publication.

[6] The revised methods of test appear in the 1960 Supplement to Book of ASTM Standards, Part 8.

Acetone in Methyl Alcohol (Methanol) (**D 1612 – 58 T**),
Alkalinity in Acetone (**D 1614 – 58 T**),
Glycerol, Ethylene Glycol, and Pentaerythritol in Alkyd Resins (**D 1615 – 58 T**),
Copper Corrosion by Mineral Spirits (Copper Strip Test) (**D 1616 – 58 T**), and
Gas Checking and Draft Test of Varnish Films (**D 1643 – 59 T**).

Tentative Recommended Practice for:

Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (**D 822 – 57 T**).

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specifications for Refined Soybean Oil (D 1462 – 57 T):[4]

Section 2.—Change the requirement for color to read: "Color (Gardner 1953 color standards), max...6" in place of "Color (Gardner 1933 permanent color standards), max...6."

Tentative Method of Test for Oil Absorption of Pigments by Gardner-Coleman Method (D 1483 – 57 T):[4]

Section 3(a).—Add the following to the apparatus: ", or a low-form 250 ml beaker."

Tentative Method for Qualitative Tests for Rosin in Varnishes (D 1542 – 58 T):[4]

Section 3(b).—Change the first sentence to read: "(*b*) *Sulfuric Acid Reagent*.—Add 35.7 ml of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) slowly to 34.7 ml of water, and cool to room temperature."

Tentative Method of Test for Ester Value of Lacquer Solvents and Diluents (D 1617 – 58 T):[4]

Title and Section 1.—Change the word "diluents" to read "thinners."

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Methyl Ethyl Ketone (D 740 – 56):[4]

Section 2.—Change the requirement for Acidity (free acid as acetic acid) to read: "not more than 0.005 per cent by weight, equivalent to 0.047 mg of KOH per gram of sample," in place of "not more than 0.003 per cent by weight, equivalent to 0.028 mg of KOH per gram of sample."

Methods of Testing Nitrocellulose Clear Lacquers and Lacquer Enamels (D 333 – 40):[4]

Section 2 (a).—In the first sentence change the specific gravity from "8.32" to "8.312." In the second sentence change the temperature from "20 C" to "25 ± 0.1 C."

Section 9.—Replace with a reference to the Standard Method of Test for Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup (ASTM Designation: D 1200).

Method of Test for Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus (D 522 – 41):[4]

Title.—Change to read: "Method of Test for Elongation of Attached Organic Coatings with the Conical Mandrel Test Apparatus."

Section 1.—Change the word "lacquer" to read "organic."

Section 3.—In Paragraph (*a*) delete the word "lacquer" from the first sentence. In the third sentence of the Note, change the word "lacquers" to read "coatings." In Paragraph (*b*) change the word "lacquer" to read "coating" in the first and second sentences.

Section 5.—After the third sentence, add the word "(Note 1)" and add the following new Note just before the present Note, designating the latter as Note 2:

NOTE 1.—If a rapid movement of the lever arm is used so as to complete the approximate 180-deg bend in 2 or 3 sec, then something other than the elongation as defined herein is measured.

In the sixth sentence, change the words "first continuous crack starting" to read "crack farthest."

Note.—Renumber as Note 2 and change the last sentence to read:

The film thickness of the coating shall be determined by the Method for Measurement of Dry Film Thickness of Non-magnetic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Magnetic Base (ASTM Designation: D 1186).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Raw Linseed Oil (**D 234 - 58 T**), and
Boiled Linseed Oil (**D 260 - 58 T**).

Tentative Methods of Test for:

Color of Orange Shellac (**D 29 - 57 T**),
Tinting Strength of White Pigments (**D 332 - 57 T**),
Specular Gloss (**D 523 - 53 T**),
Kauri-Butanol Value of Hydrocarbon Solvents (**D 1133 - 54 T**),
Color Difference Using the Hunter Multipurpose Reflectometer (**D 1260 - 55 T**),
Viscosity Reduction Power of Hydrocarbon Solvents (**D 1311 - 54 T**),
Total Rosin Acids Content of Coating Vehicles (**D 1469 - 58 T**),
Two-Parameter 60-deg Specular Gloss (**D 1471 - 57 T**),
Knoop Indentation Hardness of Paint, Varnish and Lacquer Coatings (**D 1474 - 57 T**),
Color Difference Using the General Electric Spectrophotometer (**D 1482 - 57 T**),
Color Difference Using the Colormaster Differential Colorimeter (**D 1536 - 58 T**),
Effect of Staining Agents on Organic Finishes Used in the Transportation Industry (**D 1540 - 58 T**),
Color of Transparent Liquids (Gardner Color Scale) (**D 1544 - 58 T**),
Acidity in Lacquer Solvents and Diluents (**D 1613 - 58 T**),
Print Resistance of Varnishes (**D 1645 - 59 T**),
Tentative Method of Sampling Liquid Oils and Fatty Acids Commonly Used in Paints, Varnishes and Related Materials (**D 1466 - 57 T**), and
Tentative Method of Specifying Color by the Munsell System (**D 1535 - 58 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentative:

Standard Method of Conducting Exterior Exposure Tests of Paints on Steel (D 1014 - 51):[4]

Section 2(a).—Add the following sentence to Note 1: "The pre-rusting shall be done in the same area where the subsequent testing will be done."

Section 3.—Add the following to Paragraph (*a*): "*Procedure F.*—Roller coating, or *Procedure G.*—Curtain coating." Delete the present Paragraph (*d*) and replace with the following:

(*d*) The back and edges of all test specimens shall be painted with the same system as is being tested on the front of each panel.

NOTE.—Painting the back and edges in this manner provides considerable information on the behavior of the paint system on the reverse face.

Not more than one test shall be applied to any individual panel. For example, the plan of applying one coat of primer on the entire panel and then topcoating only the upper one-half may cause the area which corrodes first to act sacrificially to decrease the corrosion on the better protected areas.

(*e*) After the test specimens have been weathering for six to seven months, scribe the specimens and observe any corrosion from this point of damage.

Standard Methods for Measurement of Wet Film Thickness of Paint, Varnish, Lacquer, and Related Products (D 1212 - 54):[4]

Section 4(a).—Delete Note 2.

Section 9.—Delete the last sentence of Note 3.

Standard Method of Test for Acid Value of Varnishes (D 1639 - 59):[7]

Section 2(b).—Delete this section.

Section 3(c).—Add to the end of the first sentence the words "and dilute to 1 liter."

Section 5.—Delete the figure "56.1" from the formula.

Tentative Method of Test for Epoxy Content of Epoxy Resins (D 1652 - 59 T):[6]

Section 1.—Change to read: "1. This method determines quantitatively the epoxy content of epoxy resins."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (D. S. Bolley, chairman) prepared the proposed Tentative Method of Test for the Qualitative Determination of Fish Oil in Drying Oils and Drying Oil Fatty Acids, and the proposed change in Specifications D 1462, as noted earlier in the report. The subcommittee also recommended retention as tentative of Specifications D 234, D 260, and D 1466 pending the completion of reviews of each which are under way. The subcommittee is continuing its thoroughgoing revision of the Standard Methods of Testing Drying Oils (D 555 - 58) and its work on a revision of the procedure for gel time of tung oil. The subcommittee is also engaged in work to further refine the procedure for determining acid value, on a revised hydroxyl value method based on the collaborative study made by the American Oil Chemists' Society, and on a rapid iodine value method. It also has organized a new working group to study improved methods for measuring gums in linseed oil.

Subcommittee IV on Traffic Paint (W. W. Burr, chairman) is making a new approach to the over-all problem of developing a test or series of tests which will correlate the results of laboratory tests with those obtained in actual road service exposures.

Subcommittee V on Solvents (E. F. Rogers, chairman) prepared the proposed Tentative Specifications for Isobutyl Alcohol (Isobutanol), for Isobutyl Acetate (93% Minimum Ester Grade), and the Tentative Methods of Test for Water Miscibility of Isopropyl Alcohol (Isopropanol), for Water Solubility of Acetone, for Permanganate Time of Tricresyl Phosphate, and for Dilution Ratio in Cellulose Nitrate Solutions for Active Solvents, Hydrocarbon Diluents, and Cellulose Nitrates, as appended to this report.[2] The subcommittee also prepared the proposed revision of Specifications D 740 which is being recommended for adoption as standard. The subcommittee also recommended the editorial changes in the title and scope of Method D 1617 and its concurrent advancement to standard as well as the proposed revision of Method D 1012 which has also been submitted to letter ballot of Research Division IV on Hydrocarbon Analysis of

---

[7] 1959 Supplement to Book of ASTM Standards, Part 8.

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Committee D-2 on Petroleum Products and Lubricants. Because of the need for additional work on the precision of the methods, the subcommittee recommended that Methods D 1133 and D 1613 be continued as tentative pending completion of the work. With respect to Method D 1311, the subcommittee recommended that it also be retained as tentative until completion of a survey of the industry to ascertain the continued need for and value of the method.

The subcommittee is engaged in work to broaden the present scope and applicability of two standards: in cooperation with Subcommittees X on Optical Properties and XI on Resins, work is under way to extend the range of the platinum-cobalt color standards employed in Method D 1209, and other work is under way to broaden the applicability of Method D 1364. Its cooperative work with Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials to resolve the existing differences in the specifications for benzene, toluene, and xylene is continuing. It has cooperated with Subcommittee 17 on Thermometers of Committee E-1 on Methods of Testing in writing specifications for a special series of six solvents distillation thermometers. It has established a special group to work with the General Services Administration and other Federal agencies to resolve present differences between Federal and ASTM specifications and test methods for solvents. It is continuing its work on description of the solvency properties of mineral spirits and has undertaken the development of specifications and test methods for vinyl acetate, formaldehyde, acetaldehyde, and pentaerythritol. It has prepared and submitted for publication a paper on "Cooperative Inter-Laboratory Study of ASTM Distillation Methods D 86 and D 1078."

Subcommittee VI on Definitions (F. B. Stieg, Jr., chairman) is engaged in the preparation of definitions of the terms "build," "durability," and "epoxy resin." The subcommittee has under review a group of definitions of optical terms prepared and submitted by Subcommittee X on Optical Properties. The subcommittee favors the more frequent use of the term "filmogen," but no definition is necessary because the term appears in standard dictionaries.

Subcommittee VII on Accelerated Tests for Protective Coatings (N. B. Garlock, chairman) prepared the proposed Tentative Method of Preparation of Aluminum Alloy Panels for Testing Paint, Varnish, Lacquer, and Related Products, the proposed Tentative Method of Making and Preparing Concrete and Masonry Panels for Testing Paint Finishes, and the proposed Tentative Method for Water Fog Testing appended to this report.[2] The subcommittee also prepared the proposed revision of Method D 1543 and recommended continuation as tentative of Method D 1540. The subcommittee is engaged in a thoroughgoing revision of the Standard Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 – 52). It is developing methods of preparation for painting of aluminized steel panels and of galvanized steel panels. Also, it is developing a method of test at 100 per cent relative humidity and a method for the accelerated evaluation of house paints and is engaged in the preparation and selection of improved photographic standards depicting various types of paint conditions and failures. Methods for determining resistance of coatings to perspiration, to detergents, and to cold-cracking are also being developed.

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (W. H. Madson, chairman) is completing its work on a proposed method of chemical analysis of strontium chromate pigments and has begun work on a method for chemical analysis of basic lead silicochromate and on the determination of lead at low concentrations in paint. It is also preparing a method of test for the determination of water-soluble salts by conductivity methods and is undertaking the consolidation of some 70 different published methods for the determination of per cent volatile matter. The subcommittee is also reviewing Standard Methods of Chemical Analysis of White Linseed Oil Paints (D 215 - 59) to determine whether all of the methods are up to date and whether they also apply to water paints.

Subcommittee IX on Varnish (J. C. Weaver, chairman) recommended the adoption as standard of Methods D 1545 and D 1643. It also recommended an editorial change in Method D 1542 and its adoption as standard. In the light of work currently being done on Methods D 1469, D 1544, and D 1645, the subcommittee recommended their retention as tentative. The subcommittee is actively engaged in work to widen the scope of the Standard Method of Test for Acid Value of Varnishes (D 1639 - 59) to include drying oils and perhaps resins, and on the revision of Standard Methods of Testing Liquid Driers (D 564 - 47) to incorporate several new test methods now available. Other activities include further work on color and haze of transparent liquids and the determination of rosin content, phenolic content, epoxy content and urethanes. Work is continuing on the establishment of the precision of Method D 1545, and work has been started to develop a suitably precise method of test for specific gravity.

Subcommittee X on Optical Properties (H. K. Hammond, III, chairman) prepared the proposed Tentative Method of Test for Hiding Power of Nonchromatic Paints and the proposed Tentative Recommended Practice for Visual Evaluation of Color Differences appended to this report.[2] The subcommittee also prepared the proposed revisions of Methods D 1365 and D 1495. The subcommittee has submitted to Subcommittee VI on Definitions definitions for the following terms relating to optical properties: color temperature of a source, correlated color temperature of a source, surface haze, bloom, hiding power, and contrast ratio. In addition, it is preparing definitions for 14 additional optical terms. The subcommittee is working on the evaluation of the accuracy and precision of color difference meters by means of a color aptitude test. It is also engaged in work on yellowness, the instrumental measurement of haze, and the application of color difference measuring instruments to the determination of tinting strength. It is also preparing a report on gloss measurement practices employed in the paint industry.

Subcommittee XI on Resins (M. H. Swann, chairman) prepared the proposed Tentative Methods of Test for Phthalate Ester Color of High-Gravity Glycerin, for Viscosity of Resin Solutions, for Urea Content of Nitrogen Resins, and for Hydrolyzable Chlorine Content of Liquid Epoxy Resins appended to this report.[2] The subcommittee also recommended adoption as standard of Methods D 1397, D 1597, and D 1615. Upon the recommendation of Section E on Epoxy Resins of Subcommittee XVI on Thermosetting Materials of Committee D-20 on Plastics, the subcommittee recommended an editorial change in the scope of Method D 1652; the change consists of the deletion of the word "coating," thus making the method applicable to all classes of epoxy resins. The subcommittee has undertaken work to enlarge the scope of the Standard Methods of Test for Nonvolatile Content of Resin Solutions (D 1259 - 58) to include epoxy resin solutions. The subcommittee is engaged in the development of a test method for total chlorine content of epoxy resins which is intended to be applicable to both liquid and solid types. Work continues on rosin resins, viscosity, softening point, and nonvolatile content.

Subcommittee XII on Latex and Emulsion Paints (P. T. Howard, chairman) prepared the proposed Tentative Method of Test for Efflorescence of Interior Latex Paints appended to this report.[2] The subcommittee is completing its work on a method of test for package stability of latex paints. It is also preparing definitions of paint film failures uniquely characteristic of latex paints and is developing a method of test for adhesion of latex paints to chalky surfaces. It is continuing its work on scrub resistance and freezing-and-thawing stability and has resumed work to develop a test to determine the resistance to biological spoilage of latex paints in containers.

Subcommittee XIII on Shellac (W. C. Scully, chairman) recommended continuation without revision of Method D 29 because of work now under way to revise the color method; a procedure is being investigated which does not require removal of the wax. Also, work is being done to determine whether color may be measured by means of instruments. In addition, an electrometric method for determination of acid value of orange shellac is being investigated.

Subcommittee XIV on Statistical Application (M. P. Morse, chairman) is completing its work on the following four proposed recommended procedures: for defining statistical terms involved in precision, for outlining various types of precision, for calculating precision, and for reporting precision. The subcommittee is cooperating with Committee E-11 on Quality Control of Materials in the development of experimental designs that can be used under the various circumstances present in the paint industry. It is also devising techniques for analyzing and presenting test data in a concise and meaningful manner.

Subcommittee XV on Specifications for Pigments, Dry and in Oil (C. L. Crockett, chairman) is completing a revision of the requirement on coarse particles retained on a No. 325 sieve in the Standard Specifications for Raw and Burnt Umber (D 763 - 48), for Raw and Burnt Sienna (D 765 - 58), and the Tentative Specifications for Venetian Red (D 767 - 52 T). It is also considering revision of the Standard Specifications for Red and Brown Iron Oxide Pigments (D 84 - 51). It has recommended to Subcommittee VIII on Methods of Chemical Analysis of Paint Materials that a method for the determination of ferrous iron be prepared.

Subcommittee XVII on Flash Point (H. E. Riley, chairman) is engaged in the revision of the Method of Test for Flash Point of Volatile Flammable Materials by Tag Open Cup Apparatus (D 1310 - 59 T). The work is intended to clarify the conditions of test for samples which have flash points below 40 F. The subcommittee has determined that Method D 1310 is generally acceptable to various organizations which use or specify flash points; it is continuing its investigation of the nomenclature used by such organizations to define various levels of hazard.

Subcommittee XVIII on Physical Properties of Materials (J. P. McGuigan, chairman) prepared the proposed Tentative Method of Test for Elongation of Attached Coatings of Paint, Varnish, Lacquer and Related Products with Cylindrical Mandrel Apparatus appended to this report,[2] and also prepared the proposed revision of Method D 522. The subcommittee prepared the editorial change in Methods D 1212. It also prepared the editorial change in and recommended adoption as standard of Method

D 1483. It also recommended adoption as standard without revision of Method D 1475, and recommended continuation as tentative of Method D 1474 because of work in progress to revise it to include the Tukon hardness tester. In addition, the subcommittee is continuing its work on metohds of test for hardness using the Pfund tester and the Kentron tester. Other work in progress includes the development of methods for the measurement of adhesion, of thixotropic properties of paints, and of settling. Work has also begun on a method of test for tensile strength, elongation and other properties using tensile testing apparatus. Work has been terminated as unfruitful on a method for preparing paint films using the wire-wound drawdown blade and on the development of a nondestructive method for determining film thickness of coatings on nonmagnetic bases. The subcommittee has recommended that the latter subject be considered as a research project to be sponsored by the Society.

Subcommittee XIX on Putty, Glazing and Caulking Compounds (H. L. Kelfer, chairman) is completing its work on a filter paper stain test and a flow-jig slump test for gun grade caulking; work on a method for measuring hardness on aging of such caulking is continuing. The subcommittee is continuing its work on a procedure for accelerated weathering and on methods of test for measuring initial and ultimate set, and surface dry. It has resumed its work with the Garvey die as a test of workability, and of the centrifuge and the vibrator as tests for package stability.

Subcommittee XXV on Lacquer (R. F. Buller, chairman) prepared the proposed revision of Methods D 333 referred to above and recommended its immediate adoption as standard. The subcommittee recommended adoption as standard of Method D 1211. It has transferred jurisdiction over Method D 522 to Subcommittee XVIII on Physical Properties of Materials. The subcommittee is continuing its work on a method for measuring the resistance of lacquers to printing and on an accelerated test for the evaluation of the settling and resuspension properties of flatted lacquers. It is also continuing with its development of an accelerated procedure for determining the light stability of clear and pigmented lacquers. In cooperation with Subcommittee VII on Accelerated Tests for Protective Coatings, it is engaged in work on a method to measure the resistance of organic coatings to perspiration. With respect to multicolor lacquers, the subcommittee is developing an accelerated freeze-shake stability test and a test for resistance to scrubbing. It is drafting a modern definition of "lacquer" and has initiated work for the purpose of rating the resistance of lacquer films to plasticizer migration from vinyl fabrics.

Subcommittee XXIX on Painting of Metals (A. J. Eickhoff, chairman) prepared the proposed Tentative Methods of Preparation of Hot-Dip Aluminum Surfaces for Painting appended to this report.[2] The subcommittee also prepared the proposed Tentative Method of Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting and the proposed Tentative Method for Preparation of Magnesium Alloy Surfaces for Painting, both of which were published as information in the ASTM *Proceedings*, Vol. 56 (1956). It also prepared the editorial changes in Method D 1014 and is continuing its work on a procedure for the preparation of galvanized surfaces for painting and in the establishment of a set of visual standards for use in classifying ferrous surfaces for painting.

This report has been submitted to letter ballot of the committee, which consists of 375 voting members; 190 members returned their ballots, of whom 174 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

W. A. GLOGER,
Secretary.

APPENDIX I

HISTORICAL REVIEW AND SUPPORTING DATA ON THE METHODS OF TEST FOR PHTHALATE ESTER COLOR OF HIGH-GRAVITY GLYCERIN (D 1257)

After developing the Specifications for High-Gravity Glycerin (D 1257),[1] Group G of ASTM Committee D-1, Subcommittee XI, undertook the development of a performance test based on the preparation of phthalate esters and determination of the ester colors.

TABLE I.—PHTHALATE ESTERS PREPARED AND GRADED BY COLLABORATORS.

NOTE.—Phthalate esters were prepared, in duplicate, from four glycerin samples by each of five collaborators. The instrumental color values (in Pt-Co units) were determined by each collaborator on his own instrument, using 16 by 150-mm test tubes.

Collaborator	Resin A	Resin B	Resin C	Resin D
A..............	1340	1080	735	465
B..............	2215	1900	1385	955
C..............	1185	810	560	240
D..............	1370	1130	710	400
E..............	2180	1860	1700	870

TABLE II.—REFEREE GRADING OF SAME PHTHALATE ESTERS AS IN TABLE I.

NOTE.—The phthalate esters of Table I were graded by a sixth collaborator on one instrument.

Prepared By	Resin A	Resin B	Resin C	Resin D
Laboratory A....	1455	1195	825	510
Laboratory B....	1400	1195	710	415
Laboratory C....	1585	1205	880	465
Laboratory D....	1300	1080	650	325
Laboratory E....	1670	1300	1050	350

In the early attempts, the color was compared visually with platinum-cobalt (Pt-Co) and Gardner color standards. Because of hue differences, the color values were erratic and this approach was abandoned. A study of the spectrophotometric curves of the Pt-Co series and of typical glycerin phthalate esters then suggested that instrumental color measurements at 450 mμ might lead to an empirical but useful test.

After preliminary trials, an interlaboratory study was carried out in which each collaborator prepared phthalate esters from a series of glycerin samples, measuring the light transmission spectrophotometrically at 450 mμ or on a colorimeter using a filter peaking at or near 450 mμ. Similar measurements were made on a series of platinum-cobalt standards and a calibration curve derived for Pt-Co color value versus per cent transmission. All optical transmissions were measured in 16 by 150-mm test tubes. The data of Table I show poor agreement between laboratories, but much better agreement (Table II) when the entire set of ester colors was measured by one collaborator on a single colorimeter. The major source of the variation between laboratories was traced to differences in the optical geometry of the colorimeters combined with a wide difference in the refractive indices of the resin esters and the platinum-cobalt color standards.

At this point, the method was revised

---

[1] 1958 Book of ASTM Standards, Part 8.

TABLE III.—RESULTS OF FINAL ROUND-ROBIN ON PHTHALIC-GLYCERIDE ESTER COLORS.

NOTE.—Phthalic-glyceride esters were prepared from four samples of glycerin (A, B, C, and D) by the proposed method. Duplicate runs were made in each of five laboratories and ester colors determined photometrically in terms of Pt-Co color units. Cuvets with plane parallel faces were used.

Glycerins	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E	Average
A-1	310	260	240	430	280	
A-2	310	260	240	430	250	
Average	310	260	240	430	265	300
B-1	1030	1100	980	1000	980	
B-2	1030	950	980	1000	990	
Average	1030	1025	980	1000	985	1005
C-1	560	580	580	560	550	
C-2	550	...	550	600	640	
Average	555	580	565	580	595	575
D-1	1420	1710[a]	1380	1400	1340	
D-2	1420	1300[a]	1470	1340	1360	
Average	1420	1505	1425	1370	1350	1415

[a] These values exceed the precision limits for repeatability and must be considered as suspect.

TABLE IV.—RESULTS OF ROUND-ROBIN ON PLATINUM-COBALT STANDARDS.

NOTE.—Standards Nos. 600, 1200, and 1800 were prepared by each of five laboratories. The following spectrophotometric data (450 mμ; Beckman DU or equivalent spectrophotometer) were obtained on all five sets by each of six collaborating laboratories and show remarkably good agreement. Cuvets with plane parallel faces were used.

Pt-Co Standard	Standard Prepared by	Absorbance Index[a] Measured by						
		Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E	Laboratory F	Average
No. 600	Laboratory A	0.160	0.160	0.166	0.169	0.175	0.172	0.167
	Laboratory B	0.160	0.167	0.161	0.165	0.166	0.172	0.165
	Laboratory C	0.160	0.169	0.163	0.167	0.173	0.168	0.167
	Laboratory D	0.159	0.158	0.158	0.166	0.163	0.164	0.161
	Laboratory E	0.157	0.162	0.157	0.166	0.161	0.157	0.160
	Average	0.159	0.163	0.161	0.167	0.168	0.167	0.164
No. 1200	Laboratory A	0.319	0.339	0.322	0.323	0.320	0.330	0.326
	Laboratory B	0.317	0.333	0.323	0.319	0.331	(0.348)	0.325
	Laboratory C	0.316	0.345	0.324	0.328	0.328	0.325	0.328
	Laboratory D	0.319	0.326	0.318	0.325	0.330	0.328	0.324
	Laboratory E	0.316	0.331	0.316	0.319	0.321	0.321	0.320
	Average	0.317	0.335	0.321	0.323	0.326	0.326	0.325
No. 1800	Laboratory A	0.482	0.500	0.481	0.484	0.478	0.498	0.487
	Laboratory B	0.480	0.506	0.482	0.480	0.484	0.513	0.489
	Laboratory C	0.479	0.502	0.489	0.483	0.494	0.495	0.490
	Laboratory D	0.480	0.498	0.480	0.482	0.478	0.493	0.485
	Laboratory E	0.480	0.499	0.479	0.480	0.479	0.484	0.484
	Average	0.480	0.501	0.482	0.482	0.483	0.497	0.487

[a] As 1-cm cells were used by all collaborators, these values also represent the optical densities.

to provide for making all color measurements in cuvets with plane parallel faces. Other refinements in the details of preparing the phthalate esters were also incorporated and another round-robin was carried out. Table III shows that agreement within and between laboratories was improved significantly. As the final step in appraising the reliability of the method, an interlaboratory study was conducted in which each collaborator prepared a series of platinum-cobalt standards and circulated them to all of the collaborators for spectrophotometric measurements. The data of Table IV show remarkably good agreement and further substantiate the usefulness of the proposed method.

The proposed tentative method is of further interest because it incorporates instructions for the preparation of platinum-cobalt standards covering the range of 200 to 2000 Pt-Co color units, which extend the Method of Test for Color of Clear Liquids (Platinum-Cobalt Scale) (D 1209),[1] beyond the present 500 maximum covered therein. The procedure for preparing these standards and the transmission data of Table IV should prove useful to other groups considering the use of much denser standards.

APPENDIX II.—EXPERIMENTAL DATA ON METHOD OF TEST FOR UREA CONTENT OF NITROGEN RESINS, PER CENT.

UREA RESIN

	Manufacturer X (23.8 per cent Expected)				Manufacturer Y (24.7 per cent Expected)				Manufacturer Z (23.6 per cent Expected)			
	Lab. B	Lab. D	Lab. E	Lab. F	Lab. A	Lab. C	Lab. E	Lab. F	Lab. A	Lab. B	Lab. C	Lab. D
Run 1 (a)	23.8	24.6	24.2	24.4	25.2	25.3	24.8	24.3	23.6	22.9	22.7	24.4
Run 1 (b)	23.5	24.6	24.4	24.1	24.9	25.0	24.7	24.7	23.7	22.9	23.2	24.1
Run 1 Average	23.6	24.6	24.3	24.2	25.0	25.2	24.8	24.5	23.6	22.9	23.0	24.2
Run 2 (a)	23.5	24.5	25.0	24.4	25.0	24.7	25.0	24.3	23.6	22.7	23.5	23.8
Run 2 (b)	23.9	24.3	24.0	24.1	24.9	25.0	24.9	24.6	23.5	22.8	23.5	23.8
Run 2 Average	23.7	24.4	24.5	24.2	25.0	24.8	25.0	24.4	23.6	22.8	23.5	23.8
Grand Average	23.7	24.5	24.4	24.2	25.0	25.0	24.8	24.5	23.6	22.8	23.2	24.0

MIXED UREA AND MELAMINE RESIN

	Manufacturer X (11.9 per cent Expected)				Manufacturer Y (12.4 per cent Expected)				Manufacturer Z (11.8 per cent Expected)			
	Lab. B	Lab. D	Lab. E	Lab. F	Lab. A	Lab. C	Lab. E	Lab. F	Lab. A	Lab. B	Lab. C	Lab. D
Run 1 (a)	12.5	12.7	12.0	11.7	12.8	12.8	12.2	12.3	11.9	11.6	11.7	12.3
Run 1 (b)	12.0	12.5	12.7	12.4	12.8	12.7	12.1	12.8	12.0	11.5	12.0	12.2
Run 1 Average	12.2	12.6	12.4	12.0	12.8	12.8	12.2	12.6	12.0	11.6	11.8	12.2
Run 2 (a)	12.0	12.5	12.0	12.1	12.7	12.3	11.7	12.4	11.9	11.4	11.9	12.0
Run 2 (b)	12.0	12.4	12.9	12.1	12.6	12.5	12.4	12.7	11.9	11.6	11.9	12.0
Run 2 Average	12.0	12.4	12.4	12.1	12.6	12.4	12.0	12.6	11.9	11.5	11.9	12.0
Grand Average	12.1	12.5	12.4	12.1	12.7	12.6	12.1	12.6	11.9	11.5	11.9	12.1

MELAMINE RESIN

	Manufacturer X (0 per cent Expected)				Manufacturer Y (0 per cent Expected)				Manufacturer Z (0 per cent Expected)			
	Lab. B	Lab. D	Lab. E	Lab. F	Lab. A	Lab. C	Lab. E	Lab. F	Lab. A	Lab. B	Lab. C	Lab. D
Run 1 (a)	0.4	0.5	...	0.0	0.3	0.4	0.3	−0.1	0.2	0.3	0.4	0.5
Run 1 (b)	0.4	0.5	0.4	−0.2	0.3	0.4	0.1	−0.4	0.3	0.4	0.3	0.4
Run 1 Average	0.4	0.5	0.4	−0.1	0.3	0.4	0.2	−0.2	0.2	0.4	0.4	0.4
Run 2 (a)	0.3	0.5	0.6	0.1	0.1	0.4	0.0	−0.7	0.1	0.2	0.1	0.3
Run 2 (b)	0.3	0.5	0.5	0.1	0.1	0.4	0.1	−0.6	0.1	0.3	0.1	0.3
Run 2 Average	0.3	0.5	0.6	0.1	0.1	0.4	0.0	−0.6	0.1	0.2	0.1	0.3
Grand Average	0.4	0.5	0.5	0.0	0.2	0.4	0.1	−0.4	0.2	0.3	0.2	0.4

APPENDIX III

RESULTS OF AN INTERLABORATORY STUDY ON VISCOSITY OF RESIN SOLUTIONS

The results of an interlaboratory study, which is the basis of the precision values given in Section 8, are given in the following table.

VISCOSITY OF RESIN SOLUTIONS, ROUND-ROBIN, MAY, 1959.
(ASTM Committee D-1, Subcommittee XI, Group A, 3, 4)

Collaborator	Bubble Time, sec									
	Standard Viscosity Liquid (Hercolyn)		Pentalyn 833 Solution, 60 per cent Solids in Toluene							
			Hot-Cut Solution				Cold-Cut Solution			
	Day 1	Day 2	Day 1		Day 2		Day 1		Day 2	
	Tube 1	Tube 1	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2	Tube 1	Tube 2
	35.2	36.0	35.3	37.4	33.9	33.9	36.2^a	36.5^a	37.0^a	36.4^a
A.............	34.9	35.8	35.1	37.5	33.8	33.9	36.4^a	35.8^a	37.2^a	36.6^a
	35.2	36.0	35.2	37.0	33.8	33.9	36.3^a	36.2^a	37.0^a	36.5^a
Average.........	35.1	35.9	35.2	37.3	33.8	33.9	36.3^a	36.2^a	37.1^a	36.5^a
	34.8	34.6	36.0	36.0	35.0	35.0	32.0	31.0	33.4	33.0
B.............	34.8	34.6	36.0	35.5	36.0	35.0	31.0	31.0	33.2	32.8
	34.8	34.8	35.0	35.0	35.4	35.2	31.5	32.0	33.0	32.6
Average.........	34.8	34.7	35.4	35.4	35.5	35.1	31.5^b	31.3^b	33.2^b	32.8^b
	35.4	34.9	35.8	35.7	35.8	35.2	36.0	36.3	36.4	35.7
C.............	35.6	35.2	35.9	35.6	35.7	35.2	35.8	36.7	36.6	35.9
	35.6	35.0	36.0	35.4	35.8	35.4	35.7	36.8	36.6	35.6
Average.........	35.5	35.0	35.9	35.6	35.8	35.3	35.8	36.6	36.5	35.7
	37.2^a	37.3^a	36.0		36.0		36.5		36.5	
	37.5^a	37.0^a	35.8		36.5		37.3		36.5	
D.............			36.5		37.0		35.5		37.0	
			36.8		36.0		36.5		36.0	
			36.0		37.0		37.2		36.7	
Average.........	37.4^a	37.2^a	36.2		36.5		36.6		36.5	
	35.5	35.4	35.1	35.2	34.3	34.2	36.5	36.4	35.2	35.1
E.............	35.6	35.5	35.1	35.2	34.4	34.2	36.4	36.4	35.2	35.1
	35.5	35.5	35.1	35.1	34.3	34.3	36.4	36.5	35.1	35.0
Average.........	35.5	35.5	35.1	35.2	34.3	34.2	36.4	36.4	35.2	35.1
Range of All.....	2.8	2.6	2.4		3.2		1.8		2.2	

---

[a] Recheck results.
[b] Values omitted from range.

REPORT OF COMMITTEE D-2
ON
PETROLEUM PRODUCTS AND LUBRICANTS*

This report contains the recommendations of the subordinate groups of Committee D-2 on Petroleum Products and Lubricants. The recommendations have been approved by Committee D-2 in accordance with the regulations of the Society and are presented herewith:

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the proposed Emergency Method of Test for Ultraviolet Absorptivity of Waxes. The emergency method was accepted by the Standards Committee on March 23, 1960, and has been assigned the designation ES 50; it is available as a separate reprint.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The following twelve proposed methods are recommended for publication as information or for republication as information:[1]

- Appendix I. Proposed Method of Test for Effect of Air Supply on Smoke Density from the Burning of Distillate Fuel Oils.
- Appendix II. Proposed Method of Test for Tetraethyllead in Gasoline by Flame Photometer.
- Appendix III. Proposed Method of Test for Naphthenes in Reformer Charge and Product by Refractivity Intercept.
- Appendix IV. Proposed Method of Test for Depentanization of Gasoline and Naphthas.
- Appendix V. Proposed Method of Test for the Isolation of Representative Saturates Fraction from Low-Olefinic Petroleum Stocks.
- Appendix VI. Proposed Method of Test for Sulfate Content of Oil-Soluble Sulfonates.
- Appendix VII. Proposed Centrifuge Method for Preparing Specimen Grids for Use in the Study of Diesel Fuels by Electron Microscopy, including an Electron Micrograph Chart.
- Appendix VIII. Proposed Description of a Sub-routine for the Extraction of a Multiplier from Table 6, D 1250 - IP/200, by Automatic Digital Computer.
- Appendix IX. Proposed Method of Test for Autoignition Temperature of Liquid Petroleum Products.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto[2] or as otherwise indicated:

Tentative Methods of Test for:

Oil Separation from Lubricating Grease During Storage,
Rust Preventive Properties of Lubricating Greases,
Functional Life of Ball Bearing Greases,
Water and Sediment in Fuel Oils by Centrifuge,
Water in Liquid Petroleum Products by Karl Fischer Reagent,
Analysis of Commercial Butane-Butene Mixtures (Gas Chromatography Method),
Calculation of Absolute Viscosity,

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The proposed methods appear in the compilation of ASTM Standards on Petroleum Products and Lubricants, October, 1960, Vol. I.

[2] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 7.

Luminometer Numbers of Aviation Turbine Fuels, as published in Appendix VIII, 1959 Report of Committee D-2, with the following revisions:

Section 7.—In line 18 of Paragraph (*b*), change the sentence to read: "Raise the wick until a meter reading approximately 5 units higher is reached."

Change Paragraph (*d*) to read as follows:

"(*d*) Run two samples of ASTM reference fuel grade *iso*octane as described in Paragraphs (*a*) to (*c*), but obtain four data points, two below and two above the rating level established with tetralin for the instrument. (The four data points should be uniformly separated by approximately 10 luminosity units.) One of the samples should be run before running the unknown test fuel; the other sample should be run after running the test fuel. Plot two curves, and at the rating level, find the lamp temperature rise for each sample of *iso*octane (Fig. 4). Average these values."

In Paragraph (*e*) change the second sentence to read: "Use this value and the average ΔT's of tetralin and of *iso*octane to calculate the Luminometer Number of this test fuel."

Note 4.—Change to read: "NOTE 4.—If a number of unknown test fuels are to be tested, the second sample of *iso*octane may be run after all of the unknown test fuels, provided all of these runs are made the same day."

Section 8. Change to read: "... where the ΔT's for the tetralin and *iso*octane are the average values obtained at the rating level of tetralin."

Section 9.—Number the Precision section as 9. Delete the second sentence and replace with: "See the Appendix to this method."

Appendix.—Add the following as an Appendix to this method:

SUMMARY OF RESULTS OF COOPERATIVE TESTS

The results of cooperative tests on this method, and a statistical analysis of the data, are given in Table I.

TABLE I.—SUMMARY OF RESULTS OF COOPERATIVE TESTS ON THE METHOD OF TEST FOR LUMINOMETER NUMBERS OF AVIATION TURBINE FUELS.

Laboratory	Luminometer Number		
	Sample 1	Sample 2	Sample 3
A.	47.9	94.8	143.0
	48.3	99.6	148.0
	48.1	94.3	143.7
	47.1	97.9	143.5
Average	47.8	96.6	144.6
B.	52.2	101.0	143.3
	56.6	96.8	144.5
	52.0	102.5	145.5
	55.4	99.0	133.0
Average	54.0	99.8	141.6
C.	48.2	94.0	138.3
	49.8	95.5	140.8
	51.3	99.3	139.5
	49.0	95.2	137.4
Average	49.6	96.0	139.0
D.	50.2	98.1	141.8
	50.9	95.0	143.1
	47.2	90.2	141.3
	50.6	101.3	140.3
Average	49.7	96.2	141.6
E.	47.2	93.7	134.3
	48.5	96.1	137.1
	47.7	94.4	140.5
	48.3	95.3	140.5
Average	47.9	94.9	138.1

Summary[a]	Sample 1	Sample 2	Sample 3
N	20	20	20
$\bar{X}$	49.8	96.7	140.7
Sr	1.4	3.9	3.2
r	4.1	8.6	9.5
S_R	2.6	3.0	3.6
R	7.7	8.9	10.6

[a] The symbols used in this summary are as follows:

N = number of individual determinations averaged,

$\bar{X}$ = grand average,

Sr = repeatability standard deviation =

$$\sqrt{\frac{\Sigma(X_1 - \bar{X}_1)^2 + \Sigma(X_2 - \bar{X}_2)^2 \cdots\cdots + \Sigma(X_K - \bar{X}_K)^2}{(N_1 - 1) + (N_2 - 1) \cdots\cdots + (N_K - 1)}}$$

where:
K operators submit data and operator i makes N_i determinations of which the mean is $\bar{X}_i$ with any individual result X_i,

r = repeatability (95 per cent probability level),

S_R = reproducibility standard deviation =

$$\sqrt{\frac{\Sigma(X_1 - \bar{X})^2}{N - 1}}, \text{ and}$$

R = reproducibility (95 per cent probability level).

Refractive Index of Viscous Materials, as published in Appendix X, 1957 Report of Committee D-2, with the aforementioned change in title and the following other revisions:

Note 5.—Delete and replace with the following:

12. *Precision.*—The following criteria should be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by more than 0.00007 units.

(*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 0.0006 units.

Section 10 (*e*).—In the last sentence put a period after "sample" and delete the last line.

Rust Protection by Metal Preservatives in the Humidity Cabinet, as published in Appendix VII to the 1959 Report of Committee D-2, with the following revisions:

Section 4 (*b*).—Add the phrase "... Type 1 (100 F min flash)."

Section 6.—To Paragraph (*a*) add the phrase "(swabbing is permissible)" after "naphtha" in the first sentence. In Paragraph (*b*), Item (*3*) delete "only" in the first sentence. In line 7, change "A10(*c*)" to "A10.(*a*) (*3*)." In Paragraph (*g*) in the second sentence, change "(*e*)" to "(*f*)."

Section 7.—In line 23 of Paragraph (*b*) change the drain time to "2 hr ± 20 min." In Paragraph (*d*), Item (*2*), after "3 hr," insert "(Preferably 6 to 8 hr to permit checking of operating conditions during these openings.)"

Note 7.—In the last line, insert "±1" after "24."

Section A.5.—In sentence 7, insert the word "on" so that the sentence reads "... is accidentally left on after warmup."

Section A.7.—After the last sentence, add the sentence: "These stones should be inspected periodically to detect possible plugging."

Section 8.—Add Item (*3*) to read: "(*3*) *Interpretation.*—The above pass and fail criteria are offered only for guidance. They are suggested as suitable for typical applications, and may be used except in cases where other pass-fail criteria are specified."

Section 10.—Add a new Section 10 to read as follows:

10. *Precision.*—This procedure is believed to represent best available practice. Operational procedures that might affect precision have been defined as closely as appears practicable. Reproducibility and repeatability data on this method are currently being determined and will be offered for inclusion in subsequent issues of the method.

ASTM Tables for Positive Displacement Meter Prover Tanks

Text for the proposed tentative tables and the method of computing them are appended hereto.[2] The complete tables are not reproduced in the report but will be published as a separate ASTM publication if this is accepted as tentative.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

D 96 – 59 T,[3] Tentative Method of Test for Water and Sediment by Centrifuge:

The proposed revision is appended hereto,[4] and includes changes in the title, scope, apparatus (a new centrifuge tube), and precision. Also, a new section describing the use of solvents has been added.

D 217 – 52 T,[5] Tentative Method of Test for Cone Penetration of Lubricating Grease:

The proposed revision, and proposed alternate method that permits higher grease penetration, are appended hereto.[6]

D 439 – 58 T,[5] Tentative Specifications for Gasoline:

Table I.—Under "Research Method Octane Number,[b] min" for "Type A" and "Type B" Gasoline, change "85 or 94[c]" to read: "87 or 96.[c]"

Footnote c.—In the last line, change "Winter 1958–1959" to "Summer 1959" and "Petroleum Products Survey No. 5" to "Petroleum Products Survey No. 12." Add the following to the footnote:

---

[3] 1958 Book of ASTM Standards, Parts 6, 7, and 10.

[4] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Parts 6, 7, and 10.

[5] 1958 Book of ASTM Standards, Part 7.

[6] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 7.

> However gasolines sold in the following areas may have antiknock values three octane numbers less than those specified above to allow for differences in altitude: the states of Idaho, Montana, Wyoming, Nevada, Utah, Colorado, New Mexico, the Arizona counties of Coconino, Navajo, Apache, Yavapai, Gila, Graham, Greenlee, Cochise and Santa Cruz; that part of South Dakota west of and including the counties of Perkins, Meade, Pennington, Jackson, Mellette and Todd; that part of Nebraska west of and including the counties of Cherry, Thomas, Logan, Lincoln, Frontier and Redwillow; that part of Kansas west of and including the counties of Decatur, Sheridan, Gove, Lane, Finney, Gray and Meade; the Oklahoma counties of Cimarron, Texas and Beaver; and that part of Texas west of and including the counties of Lipscomb, Hemphill, Wheeler, Collingsworth, Hall, Motely, Dickens, Kent, Scurry, Mitchell, Glasscock, Reagan, Crockett, and Terrell.

TABLE II.—SPECIFICATIONS FOR ASTM KNOCK TEST REFERENCE FUELS.

	ASTM *Iso*octane	ASTM *n*-Heptane	ASTM Blends
ASTM Motor Octane Number[a]	100 ± 0.1	0.0 ± 0.2	nominal ±0.1
Density at 20 C,[b] g per ml	0.69193 ± 0.00015	0.68380 ± 0.00015	
Refractive Index,[c] N_D^{20C}	1.39145 ± 0.00015	1.38770 ± 0.00015	
Freezing Point,[d] deg Cent	−107.442 min	−90.710 min	
Distillation:[e]			
50 per cent Recovered, deg Cent (at 760 mm of Hg)	99.238 ± 0.025	98.427 ± 0.025	
Differential, 80 per cent Recovered minus 20 per cent Recovered, deg Cent	0.020 max	0.020 max	
TEL Content,[f] ml per gal	not greater than 0.002	not greater than 0.002	

[a] Determined in accordance with ASTM Method D 357, Test for Knock Characteristics of Motor Fuels Below 100 Octane Number by the Motor Method.

[b] Determined in accordance with ASTM Method D 1217, Test for Density and Specific Gravity of Liquids by Bingham Pycnometer.

[c] Determined in accordance with ASTM Method D 1218, Measurement of Refractive Index and Refractive Dispersion of Hydrocarbon Liquids.

[d] Determined in accordance with ASTM Method D 1015, Test for Freezing Points of High-Purity Hydrocarbons.

[e] For equipment and method used, see *Journal of Research*, Nat. Bureau Standards, Vol. 44, No. 3, 1950, pp. 309 and 310 (RP2079).

[f] To be determined in the certification test in accordance with ASTM Method D 1368, Test for Tetraethyllead in Trace Concentrations.

D 483 – 52 T,[5] Tentative Method of Test for Unsulfonated Residue of Petroleum Plant Spray Oils:

The proposed revision, as appended

hereto,[6] includes editorial changes and new information on precision.

D 614 – 59 T,[7] Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Aviation Method:

Section 213(a): Reference Materials.—Change to read as shown in the accompanying Table II.

D 875 – 53 T,[5] Tentative Method of Calculation for Olefins and Aromatics in Gasoline:

The proposed revision, as appended hereto,[6] includes changes in title, scope, procedure and precision in order to include a new boiling range correction, an option of analysis by cuts, and new information on precision.

D 893 – 52 T,[5] Tentative Method of Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils:

The proposed revision, as appended

---

[7] See ASTM Manual for Rating Aviation Fuels by Supercharge and Aviation Methods, 1958.

hereto,[6] includes changes in scope, reagents and procedure in order to provide for the testing of used lubricating oils containing detergents.

D 909 – 59 T,[7] Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Supercharge Method:

Section 209(a): Reference Materials.—Change to read as shown above for Method D 614.

D 1019 – 58 T,[5] Tentative Method of Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates:

Section 8.—Replace with the following:

8. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability) for 10-ml samples. (For 5-ml samples, numbers will be approximately twice as large.)

(*a*) Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts (see Note 14):

	Repeatability, per cent
Standard Flask	1.0
Precision Flask	0.6

(*b*) The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts (see Note 14):

	Reproducibility, per cent
Standard Flask	2.0
Precision Flask	2.0

NOTE 14.—Repeatability was calculated from estimates of standard deviations for the standard flask of 0.40 with 41 deg of freedom and for the precision flask of 0.18 with 38 deg of freedom.[5] For reproducibility the variances of the data for both flasks, not being significantly different, were pooled to permit calculation of reproducibility from a pooled estimate of standard deviation of 0.735 with 66 deg of freedom.[5]

[5] The boiling range, per cent olefins, and per cent aromatics of the samples used in testing by Method D 1019 are listed in the appendix published for information in 1960 with Method D 875 – 60 T. Complete data will be filed at ASTM Headquarters.

Summary of Statistical Information.—The Summary of Statistical Data in the accompanying Table III,[8] used in deriving the proposed new section on precision for Method D 1019, will be published as information in an Appendix to the method.

D 1086 – 56 T,[5] Tentative Method for Measuring the Temperature of Petroleum and Petroleum Products:

Section 4(d).—Add Item (*1*) to read:

(*1*) *Air Jacketed Thermometer.*—In the assembly illustrated in Fig. 3A, the ASTM thermometer is secured and sealed in a glass air jacket. The air jacket is enclosed and cushioned at top and bottom in a spark-resistant metal case. This assembly is suitable for temperature determinations of heated or non-heated petroleum or its products through thermometer wells in pressure tanks, or by direct immersion in non-pressure tanks. Longer immersion times may be necessary in certain instances other than those given in Table IV.

New Figure.—Include a new Fig. 3A as shown in the accompanying Fig. 1.[9]

D 1319 – 58 T,[5] Tentative Method of Test for Hydrocarbon Types in Liquid Petroleum Products (Fluorescent Indicator Adsorption (FIA) Method):

Title.—Change to read: "Tentative Method of Test for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption."

Section 3.—In Paragraph (*a*) replace the first three sentences with: "*Adsorption Columns,* with standard wall tubing, as shown on the left in Fig. 1, or with true bore tubing,[4] as shown on the right in Fig. 1, made of glass and consisting of a charger section with a capillary neck, a separator section, and an analyzer section. Draw out one end of the analyzer section of the standard column to a fine capillary to retain the gel." In the next to the last sentence, put a period after "(Note 2)" and delete the rest of the paragraph. Change Note 2 to read as follows:

---

[8] Table III is not included in this report but appears in the revised standard; see 1960 Supplement to Book of ASTM Standards, Part 7.

[9] This figure is not included in this report but appears in the revised tentative; see 1960 Supplement to Book of ASTM Standards, Part 7.

NOTE 2.—Calibrations of standard wall tubing would be impractical; however, any significant variation in the outside diameter along the tube may be taken as an indication of irregularities in the inner diameter and such tubing should not be used. For columns with true bore tubing, the inner diameter of the analyzer section shall be 1.60 to 1.65 mm and an approximately 100 mm "thread" of mercury (as measured by ordinary calipers) shall not vary by more than 0.33 mm in any part of the analyzer section. In glass-sealing the various sections to each other, long taper connections shall be made instead of shouldered connections. Support the silica gel by a short piece of capillary tubing connected by a spherical joint as shown.

Change Paragraph (*b*) to read as follows:

(*b*) *Zone Measuring Device.*—The zones may be marked with a glass-writing pencil and the distances measured with a meter rule, with the analyzer section lying horizontally. Alternatively, the meter rule may be fastened adjacent to the column. In this case, it is convenient to have each rule fitted with four movable metal index clips (Fig. 1) for marking zone boundaries and measuring the length of each zone. Very narrow zones may be marked with a pair of clips, one of which is the mirror image of the other.

Delete Note 3 and renumber subsequent notes.

Section 4.—Change Paragraph (*c*) to read as follows:

(*c*) *Fluorescent Indicator*, may be used as Dye Mixture or Dyed Gel:

(*1*) *Dye Mixture*,[7] consisting of a mixture of Sudan III with olefin and aromatic dyes dissolved in xylene. Stir or shake the mixed dyes thoroughly before each use. Store the dye mixture in a cool, dark place when not in actual use.

(*2*) *Dyed Gel*, either a standard dyed gel,[7] consisting of a mixture of recrystallized Petrol Red AB4 and purified portions of the olefin and aromatic dyes obtained by chromatographic adsorption following a definite, uniform procedure, and deposited on silica gel (Note 3); or a dyed gel prepared as follows: add to 150 ml of ether a volume of the well-mixed fluorescent indicator dye solution equivalent to 200 mg. To this mixture add 50 g of the 100- to 200-mesh gel (Paragraph (*a*)) to form a slurry. Remove the ether slowly by evaporation and distill off the xylene from the mixture at approximately 50 C and 1 mm pressure. Store the prepared gel in a dark place under a nitrogen atmosphere.

NOTE 3.—This mixture has been cooperatively tested and has been found to produce more brilliant and definite zones when applied to kerosine type fuels and is especially recommended for use in analyzing commercial turbine fuels for aircraft. Results from such use have given a precision somewhat better than stated in Section 8.

Delete Paragraph (*d*) and Note 4, relettering and renumbering subsequent paragraphs and Notes accordingly.

Section 5.—Change Paragraph (*a*) to read as follows:

(*a*) Prepare two columns for simultaneous testing. Suspend each column freely from a loose-fitting clamp placed immediately below the spherical joint of the charger section. Pour the specified 100 to 200-mesh silica gel into the column through a glass funnel until the required amount has been added (Section 5 (*c*)). While supporting the fragile analyzer lightly with the palm of the hand, vibrate or tap the column to pack the gel firmly. Rotate the column slowly by hand while vibrating or tapping to prevent horizontal segregation of particles and to avoid the possibility of channeling. Alternately vibrating racks have been used successfully for this purpose. (Packing is conveniently done by using a small electric vibrator, applying the vibrator up and down the tubing.) Add more of the 100 to 200-mesh silica gel until the tightly packed gel extends approximately 40 mm into the charger section (a 5-min vibration period is usually satisfactory when the electrical vibrator is used).

Delete Note 7, and renumber Notes 6, 8, and 9 as 4, 5, and 6.

Change Paragraph (*c*) to read as follows:

(*c*) When using dye mixture, mix it and the sample in a volume ratio of approximately 1 to 1000, using the tip of a needle to transfer the dye mixture; as little as 0.5 parts per 1000 is sufficient; above 5 parts per 1000, the xylene content of the dye mixture may alter the results. When using dyed gel, add the regular gel until the separator section is half full, then add a 3 to 5-mm layer of dyed gel. Add more regular gel and vibrate the columns until it is firmly packed.

In Paragraph (*f*), in the first sentence, replace the words "quickly mark" with ". . . ., make a set of readings by quickly marking. . . ." Add after last sentence the following: "If the boundaries have been marked off with index clips, record the measurements."

Renumber Note 10 as Note 7 and change to read as follows:

NOTE 7.—If the dye mixture or the dyed gel prepared as described in Section 4(*c*)(*2*) is used, the use of a Wratten No. 47 or No. 50 light filter between the column and the eyes may aid in the recognition of the aromatic zone front.

In Paragraph (*g*), in the first sentence, replace the words "again mark the zones" with "make a second set of readings by marking the zones." Change the last sentence to read: "If the boundaries have been marked off with index clips, record the measurements."

Renumber Note 11 as Note 8.

Section 6(*b*).—Delete and renumber Paragraph (*a*) as Section 6.

New Section.—Add a new Section 7 on Report to read as follows:

7. *Report.*—Report the per cent by volume to the nearest 0.5 per cent, adjusting the amount of the largest component, if necessary, so that the sum of the components is 100 per cent. Report as the test result the average of the four sets of observations obtained from the two successive sets of readings on each pair of columns.

Section 7.—Renumber as Section 8 and change to read as follows:

8. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

	Repeatability, per cent
Aromatics	2
Olefins	2
Saturates	2

(*b*) The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts:

	Reproducibility, per cent
Aromatics	3
Olefins	3
Saturates	3

D 1320 – 57 T,[5] Tentative Method of Test for Tensile Strength of Paraffin Wax:

Section 5.—Delete Paragraph (*b*) and Footnote 4, renumbering the subsequent footnotes accordingly.

Section 7(*d*).—Change the next to last sentence to read: "After 2 hr ± 10 min, during which time the room temperature is 73 ± 1 F and the relative humidity is 50 ± 5 per cent, carefully remove the wax specimens from the molds and place them in the water bath, maintained at 73 ± 0.5 F for 15 min, before breaking in the testing machine."

New Note.—Add a new Note 4 to follow Section 10(*b*), the new note to read: "NOTE 4.—Recent cooperative tests indicate the specified reproducibility to be questionable. This subject is being given further study."

D 1321 – 57 T,[5] Tentative Method of Test for Needle Penetration of Petroleum Waxes:

The proposed revision, as appended hereto,[6] includes changes to make the method apply to waxes of up to 150 penetration.

D 1656 – 59 T,[10] Tentative Method of Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method:

Section 214(*a*): *Reference Materials.*—Change to read as shown above for Method D 614.

---

[10] 1959 Supplement to Book of ASTM Standards, Part 7.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Recommended Practice for Volume Calculations and Corrections in the Measurement of Petroleum and Petroleum Products (**D 1087 – 57 T**)[5] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Kinematic Viscosity (**D 445 – 53 T**)[5] be approved for reference to letter ballot of the Society for adoption as standard as appended hereto.[11] The proposed changes eliminate procedure for calibration of master viscometers, provide new precision information, and include a more complete appendix describing widely used viscometers.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated in the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

D 127 – 49,[5] Standard Method of Test for Melting Point of Petrolatum and Microcrystalline Wax:

Section 2.—In the italicized heading and in the first phrase of the first sentence add the words "and microcrystalline wax" after "petrolatum." In the first sentence, replace "which petrolatum" with "which material."

Section 6.—Add a new Section 6 on Precision to read as follows:

6. *Precision.*—The following criteria should be used for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatability.*—Duplicate results by the same operator should not be considered suspect unless they differ by the following amount:

Repeatability .... 1.5 F

(*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amount:

Reproducibility .... 6.0 F

D 129 – 58,[5] Standard Method of Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method:

New Note.—Add a new Note 1 to follow Section 1, renumbering subsequent Notes accordingly; the new note to read as follows:

NOTE 1.—This method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid solutions and would interfere in the precipitation step. These interfering elements include Fe, Al, Ca, Si and Pb which are sometimes present in greases, lube oil additives or additive oils. Other acid insoluble materials that interfere are silica, molybdenum disulfide, asbestos, mica, etc. The method is not applicable to used oils containing wear metals, and lead or silicates from contamination. Samples which are excluded can be analyzed by ASTM Method D 1552.

Section 5.—In Paragraph (*d*), after the third sentence, add a sentence to read: "Add 10 ml of saturated bromine water to the washings in the 600-ml beaker." Change the present fifth sentence to read: "Place the sample cup in a 50-ml beaker, add 5 ml of saturated bromine water, 2 ml of HCl and enough distilled water to just cover the cup."

Reword the first sentence of Paragraph (*e*) to read: "Evaporate the combined washings to 200 ml on a hot plate or . . . etc." After the sixth sentence add: "(Note 5)."

Note 5.—Add a new Note 5 to follow Section 5(*e*), the new Note to read: "NOTE 5.—A weighed porcelain filter crucible (Selas type) of 5 to 9 μ porosity may be used in place of the filter paper.

---

[11] The revised method of test appears in the 1960 Supplement to Book of ASTM Standards, Part 7.

In this case the precipitate is washed free of chloride and then dried to constant weight at 500 ± 25 C." Renumber the present Notes 4 and 5 as 6 and 7, respectively.

D 357 – 59,[12] Standard Method of Test for Knock Characteristics of Motor Fuels Below 100 Octane Number by the Motor Method:

Section 214(a): Reference Materials.—Change to read as shown above for Method D 614.

D 665 – 54,[5] Standard Method of Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water:

New Method.—At the end of Section 7 add a proposed new method for testing heavier-than-water fluids, as follows:

PROPOSED METHOD FOR TESTING HEAVIER-THAN-WATER FLUIDS

8. *General.*—The stirring action provided by the stirrer prescribed in Section 3(*d*) is not sufficient to mix thoroughly the water and test sample when the fluid under test is heavier than water. The following sections define changes for testing of heavier-than-water fluids. Except as specifically stated, all the requirements of the previous Sections 1 to 7 shall apply.

9. *Apparatus:* (*a*) *Beaker Cover.*—Same as prescribed in Section 3(*c*) (Note 11).

NOTE 11.—Beaker covers and test specimen holders constructed of methyl methacrylate resins are satisfactory for occasional testing of heavier-than-water fluids. However, after prolonged contact with such fluids, methyl methacrylate resins become etched and opaque. Polyethylene or tetrafluoroethylene are unaffected by contact with such fluids and beaker covers and test specimen holders of such construction are recommended for frequent testing of heavier-than-water fluids.

(*b*) *Stirrer.*—Same as prescribed in Section 3(*d*) but with an auxiliary blade attached to the stirrer shaft. The auxiliary blade shall be of stainless steel, 18.7 by 12.5 by 0.6 mm (0.75 by 0.50 by 0.025 in.), as shown in Fig. 5 (the accompanying Fig. 2). The auxiliary blade shall be positioned on the stirrer shaft so that the bottom edge of the auxiliary blade is 57 mm (2.25 in.) above the top edge of the fixed lower blade, and so that the flat surfaces of both blades are in the same vertical plane.

10. *Test Specimen and Its Preparation.*—Same as prescribed in Section 4(*a*) (Note 11).

New Figure.—Add a new Fig. 5, as shown in the accompanying Fig. 2.[13]

D 908 – 59,[12] Standard Method of Test for Knock Characteristics of Motor Fuels Below 100 Octane Number by the Research Method:

Revise as indicated above for the proposed revision to D 357, Motor Method.

D 938 – 49,[5] Standard Method of Test for Congealing Point of Pharmaceutical Petrolatums:

Title.—Change to read: "Standard Method of Test for Congealing Point of Petrolatums and Petroleum Waxes."

Section 1.—Delete the word "pharmaceutical," and add the phrase: "and of petroleum waxes" to the end of the section.

Section 2.—Add the words "or petroleum wax" after "petrolatum."

TENTATIVES CONTINUED WITHOUT REVISION

The committee has reviewed the following tentatives that have stood one or more years without revision and, for good and sufficient reason discussed within the committee, recommends their continuation as tentatives without revision:

D 93 – 58 T, Flash Point by Pensky-Martens Closed Tester

D 94 – 56 T, Saponification Number by Color-Indicator Titration

D 156 – 53 T, Saybolt Color of Petroleum Products (Saybolt Chromometer Method)

D 270 – 55 T, Sampling Petroleum and Petroleum Products

D 285 – 54 T, Distillation of Crude Petroleum

D 286 – 58 T, Autogenous Ignition Temperatures of Petroleum Products

D 322 – 58 T, Dilution of Gasoline-Engine Crankcase Oils

D 892 – 58 T, Foaming Characteristics of Lubricating Oils

---

[12] ASTM Manual for Rating Motor Fuels by Motor and Research Methods, 1960.

[13] This figure is not included in this report but appears in the revised standard; see 1960 Supplement to Book of ASTM Standards, Part 7.

D 974 – 58 T, Neutralization Number by Color-Indicator Titration
D 1091 – 58 T, Phosphorus in Lubricating Oil and Additives
D 1092 – 58 T, Apparent Viscosity of Lubricating Greases
D 1218 – 52 T, Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
D 1367 – 55 T, Lubricating Qualities of Graphites
D 1401 – 56 T, Emulsion Characteristics of Steam-Turbine Oils
D 1403 – 56 T, Cone Penetration of Lubricating Grease Using One-Quarter Scale Cone Equipment (One-Quarter Scale of Method D 217 Cone and Worker)
D 1404 – 56 T, Deleterious Particles in Lubricating Grease
D 1406 – 56 T, Liquid Calibration of Tanks
D 1407 – 56 T, Calibrating Barge Tanks
D 1408 – 58 T, Calibrating Spherical and Spheroidal Tanks
D 1410 – 56 T, Calibrating Stationary Horizontal Tanks
D 1549 – 58 T, Zinc in Lubricating Oils and Additives (Polarographic Method)
D 1550 – 58 T, ASTM Butadiene Measurement Tables
D 1551 – 58 T, Sulfur in Petroleum Oils (Quartz-Tube Method)
D 1552 – 58 T, Sulfur in Petroleum Products (High-Temperature Method)
D 1553 – 58 T, Analysis of Graphites Used as Lubricants
D 1555 – 58 T, Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene

AMERICAN STANDARDS

The committee recommends the following for action by ASA Sectional Committee Z11 on Petroleum Products and Lubricants:

Reapproval of American Standards Revised in This Report:

Z11.3—1952, Cone Penetration of Lubricating Grease (217),
Z11.8—1959, Water and Sediment in Crude Oils by Centrifuge (D 96),
Z11.22—1949, Melting Point of Petrolatum and Microcrystalline Wax (D 127),
Z11.37—1959, Knock Characteristics of Motor Fuels Below 100 Octane Number by the Motor Method (D 357),
Z11.41—1952, Unsulfonated Residue of Petroleum Plant Spray Oils (D 483),
Z11.61—1949, Congealing Point of Petrolatums and Petroleum Waxes (D 938),
Z11.69—1959, Knock Characteristics of Motor Fuels Below 100 Octane Number by the Research Method (D 908),
Z11.71—1958, Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019), and
Z11.85—1955, Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (D 665).

Approval as American Standard:

D 445, Kinematic Viscosity,
D 1266, Sulfur in Petroleum Products and Liquefied Petroleum Gases (Lamp Method), and
D 1500, ASTM Color of Petroleum Products (ASTM Color Scale).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[14]

This report has been submitted to letter ballot of the committee which consists of 143 voting members; 90 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. M. SMITH,
Chairman.

W. T. GUNN,
Secretary.

---

[14] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specification for:

Fuel Oils (D 396 – 48 T)

Revision of Tentative Methods of Test for:

Thermal Stability of Aviation Turbine Fuels (D 1660 – 59 T)

Revision of Tentative Classification of:

Diesel Fuel Oils (D 975 – 59 T)

These recommendations were accepted by the Standards Committee on September 29, 1960, and the revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 7.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS*

Committee D-3 on Gaseous Fuels held no meetings during the year; however, meetings of Subcommittees III on Determination of Calorific Value of Gaseous Fuels and VII on Complete Analysis of Chemical Composition of Gaseous Fuels were held on June 22, 1959, in conjunction with the ASTM Annual Meeting in Atlantic City, N. J. Both these subcommittees are preparing new or revised methods for publication as tentative.

The officers elected for the ensuing term of two years are as follows:

Chairman, D. V. Kniebes.
Vice-Chairman, F. E. Vandaveer.
Secretary, K. R. Knapp.
Advisory Committee: D. V. Kniebes, chairman, W. M. Cline, Jr., E. W. Evans, A. W. Gauger, E. G. Hammerschmidt, K. R. Knapp, and F. E. Vandaveer.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Measurement of Gaseous Samples (H. B. McNichols, chairman).—Three papers dealing with measurement of small volumes were presented at technical sessions of the ASME Research Committee on Fluid Meters at its December, 1959, meeting. These papers indicated encouraging results with small flows although several design features are critical. Distribution of these papers to members of the subcommittee for study and review at a later meeting is planned.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (A. F. Cascioli, chairman) agreed at its June meeting on details of a proposed tentative procedure for heating value determination by the recording calorimeter. It is based on results of the American Gas Assn's. sponsored research by the National Bureau of Standards on the accuracy of the Cutler-Hammer recording calorimeter on gases of high Btu, and will cover the range of 900 to 1200 Btu per cu ft. Preparation of the method is well along and early distribution of a tentative draft is planned for discussion at a meeting scheduled for June 27.

Subcommittee V on Determination of Special Constituents of Gaseous Fuels (O. W. Lusby, chairman) has exchanged correspondence with others interested in the development of a method for the determination of hydrogen sulfide in concentrations of one grain or less per 100 cu ft. Consideration is being given to a method based on the use of methylene blue or to one using gas chromatography.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (D. V. Kniebes, chairman) is preparing two analytical methods. A proposed revision of the Tentative Method for Analysis of Carbureted Water Gas by the Mass Spectrometer (D 1302 – 53 T) was distributed to its members prior to the June 22, 1959, meeting and also to Subcommittee IV on Methods, Data, and Nomenclature of Committee E-14 on Mass Spectrometry. Further changes to cover comments received are well advanced. A meeting is scheduled for

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

June 27 to discuss the proposed changes. At that time it is hoped also to have available for discussion a draft of a new proposed tentative for analysis of natural gas by gas chromatography. A procedure has been written and is awaiting laboratory test.

This report has been submitted to letter ballot of the committee, which consists of 35 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. V. KNIEBES,
Chairman.

K. R. KNAPP,
Secretary.

REPORT OF COMMITTEE D-4
ON
ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the year: in Atlantic City, N. J., on June 26, 1959, and in Chicago, Ill., on Feb. 5, 1960.

The committee has unanimously elected Walter J. Emmons as an Honorary Member of the committee.

The committee sponsored two symposia during the 1959 Annual Meeting: one on "The Practical and Statistical Significance of Tests and Properties of Bituminous Paving Binders," and the other on "Methods of Test for Design of Bituminous Paving Mixtures," as well as a general session on Road and Paving Materials.[1] During the Third Pacific Area National Meeting held in San Francisco, the committee sponsored two papers sessions on Oct. 15, 1959. Seven technical papers were presented in these two sessions.[2]

The committee took action to abolish the Joint Subcommittee on Tests and Specifications for Stabilized Soils of Committees D-4 and D-18 on Soils for Engineering Purposes. The functions of this joint subcommittee are now solely under the jurisdiction of Committee D-18.

The officers elected for the ensuing term of two years are as follows:

Chairman, A. B. Cornthwaite.
First Vice-Chairman, R. E. Bollen.
Second Vice-Chairman, J. E. Gray.
Third Vice-Chairman, J. L. Wilks.
General Secretary, B. F. Kallas.
Membership Secretary, D. W. Lewis.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and specifications as appended hereto:[3]

Tentative Method of Test for:

Effect of Heat and Air on Asphaltic Materials (Thin-Film Oven Test).

Tentative Specifications for:

Preformed Expansion Joint Fillers for Concrete Paving and Structural Construction (Non-Extruding and Resilient Bituminous Types),
Preformed Expansion Joint Fillers for Concrete Paving and Structural Construction (Non-Extruding and Resilient Nonbituminous Types), and
Hot-Mixed, Hot-Laid Tar Paving Mixtures.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[3] of the Tentative Method of Test for Resistance to Plastic Flow of Bituminous Mixtures by Means of the Marshall Apparatus (**D 1559 – 58 T**)[4] and continuation of the method as tentative.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The papers presented at these symposia and the general session have been published as *ASTM STP No. 252.*

[2] To be issued as separate publication *ASTM STP No. 277.*

[3] The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 4.

[4] 1958 Book of ASTM Standards, Part 4.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revision as follows of the Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8 – 55):[4]

Change the definition of Free Carbon in Tars to read: "The hydrocarbon fraction which is precipitated from a tar by dilution with carbon disulfide or benzene."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Compressive Strength of Bituminous Mixtures (**D 1074 – 58 T**),[4] and

Moisture or Volatile Distillates in Bituminous Paving Mixtures (**D 1461 – 57 T**).[4]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of the Standard Methods of Testing Emulsified Asphalts (**D 244 – 55**),[4] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society:

Section 18(c).—Add the following sentence before the last sentence: "Continue kneading the lump for another 2 min after the addition of the $CaCl_2$ solution."

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards:

Standard Specifications for:

Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surface Courses (**D 947 – 55**), and

Asphaltic Mixtures for Sheet Asphalt Pavements (**D 978 – 54**).

Standard Method of Test for:

Testing Preformed Expansion Joint Fillers for Concrete (Non-Extruding and Resilient Types) (**D 544 – 49**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following methods and specifications:

Tentative Methods of Test for:

Vacuum Distillation of Liquid and Semi-Solid Asphaltic Materials to Obtain a Residue of Specified Penetration (**D 1189 – 52 T**),

Resistance to Deformation and Cohesion of Bituminous Mixtures by Means of Hveem Apparatus (**D 1560 – 58 T**),

Preparation of Test Specimens of Bituminous Mixtures by Means of California Kneading Compactor (**D 1561 – 58 T**),

Loss on Heating of Oil and Asphaltic Compounds (**D 6 – 39 T**),

Testing Concrete Joint Sealers (**D 1191 – 52 T**), and

Scratch Hardness of Coarse Aggregate Particles (**C 235 – 57 T**).

Tentative Revision of Method of Test for:

Emulsified Asphalts (**D 244 – 55**).

Tentative Specifications for:

Asphalt Cement for Use in Pavement Construction (**D 946 – 47 T**),

Crushed Stone, Crushed Slag, and Crushed Gravel for Dry-Bound or Water-Bound Macadam Base Courses (**D 694 – 57 T**),

Mineral Filler for Sheet Asphalt and Bituminous Concrete Pavements (**D 242 – 57 T**), and

Concrete Joint Sealer, Hot-Poured Elastic Type (**D 1190 – 52 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Definitions of Terms Relating to Materials for Roads and Pavements (D 8 – 55):[4]

In the first definition, change the term "Bitumens" to "Bitumen."

Standard Method of Test for Distillation of Cut-Back Asphaltic Products (D 402 - 55):[4]

Section 3(g).—Change to require thermometer 8F instead of thermometer 3F.

Standard Method of Test for Bitumen Content of Paving Mixtures by Centrifuge (D 1097 - 58):[4]

Footnote 4.—Delete this footnote.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A-2 on Nomenclature. (J. W. Donegan, chairman) is considering substantial revisions and editing of Definitions D 8.

Subcommittee A-4 on Papers (N. W. McLeod, chairman) sponsored a general papers session on road and paving materials during the 1959 Annual Meeting.

Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) is continuing its studies of mechanical stability test methods for bituminous mixtures. Many revisions are being made in Method D 1559, commonly known as the Marshall test, to improve both the accuracy and the utility of this test. Similarly, Method D 1560, commonly known as the Hveem test, is being studied by a special group known as the Triaxial Institute on the West Coast for improvements with the objective of advancing the method to standard. This committee is planning a Manual of Recommended Practices on Design of Bituminous Paving Mixtures.

Subcommittee B-5 on Softening Point (D. F. Fink, chairman) is preparing a new method of test by ring and ball apparatus for asphalts and tar pitches using a single bath and single thermometer, complete with precision statement.

Subcommittee B-6 on Extraction and Recovery of Constituents from Bituminous Mixtures (E. W. Klinger, chairman) has prepared four methods of test involving the extraction of bituminous materials from aggregates.

Subcommittee B-17 on Emulsion Tests (P. E. McCoy, chairman) is preparing a new method of test for stone coating and water resistance of coarse aggregate mixing asphalt emulsions. The committee is also active in studying Method D 244 for improvements in precision and techniques.

Subcommittee B-19 on Accelerated Tests for Durability of Bituminous Materials (D. C. Taylor, chairman) prepared the proposed Tentative Method of Test for Effect of Heat and Air on Asphaltic Materials (Thin-Film Oven Test) appended to this report.[3] A precision statement has now been prepared and submitted to letter ballot of the committee. The subcommittee is continuing its work on the sliding plate microviscometer techniques for determining viscosity and aging index of bituminous materials.

Subcommittee B-24 on Penetration Test (J. Y. Welborn, chairman) is planning cooperative testing to determine the precision of Method D 5. It is also planning to study precision of the penetration of the residue from the thin film oven test.

Subcommittee B-26 on Effect of Water on Bituminous Coated Aggregates (W. H. Goetz, chairman) has five active task forces studying various problems in connection with coating and adhesion of bitumen to aggregates.

Subcommittee C-1 on Asphalt Cements (A. R. Curtis, chairman) is giving consideration to possible revisions of Specification D 946.

Subcommittee C-4 on Emulsified

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Asphalts (C. E. Proudley, chairman) has under consideration a number of revisions of Specification D 977.

Subcommittee C-10 on Plant-Mix Bituminous Surfaces and Bases (Fred Hubbard, chairman) has been quite active in improving specifications for bituminous paving mixtures and for aggregates used in such mixtures. The activities have resulted in an entirely new Tentative Specification for Hot-Mixed, Hot-Laid Asphalt Paving Mixtures (D 1663 – 59 T), replacing two former specifications: Specification for Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surface Courses (D 947 – 55) and Specification for Asphaltic Mixtures for Sheet Asphalt Pavements (D 978 – 54).

A new Tentative Specification for Hot-Mixed, Hot-Laid Tar Paving Mixtures, appended to this report,[3] is being recommended to the Society as a result of the work by this subcommittee. Several revisions were made in Specifications D 692 and D 1073.

Subcommittee C-12 on Bituminous Surface Treatments (J. O. Izatt, chairman) is engaged in a review of Specification D 1139 with the view of proposing modifications of this specification.

Subcommittee C-13 on Bituminous Paving Plant Operations (J. E. Ward, chairman) has under consideration a number of proposed changes in Specifications D 995.

Subcommittee C-14 on Standard Sizes of Coarse Aggregates for Highway Construction (Harold Allen, chairman) has made a thorough study of aggregate gradations contained in Specifications D 448 and has concluded that this specification is adequate for the present time.

Subcommittee D-3 on Joint Fillers and Asphalt Planks (D. O. Woolf, chairman) has under consideration proposed specifications and methods for cold applied sealers of the single or multiple component type and for hot or cold applied jet fuel resistant sealers.

This report has been submitted to letter ballot of the committee, which consists of 189 members; 123 members returned their ballots, of whom 114 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. B. Cornthwaite,
Chairman.

John M. Griffith,
General Secretary.

REPORT OF COMMITTEE D-5
ON
COAL AND COKE*

Committee D-5 on Coal and Coke held one meeting during the year in Atlantic City, N. J., on June 25, 1959. Two meetings of the Advisory Subcommittee were held in Atlantic City, N. J., on June 22, 1959, and in Chicago, Ill., Feb. 3, 1960. Subcommittees and task groups held meetings in Atlantic City in June 1959, and in Chicago and New York City in February 1960.

The committee was saddened by the sudden death in September 1959 of Mr. B. P. Mulcahy, Chairman of Subcommittee XXII and an active member over many years. During the year the committee lost 4 members, and 7 members were added. The present 65 voting members are classified as: producers, 18; consumers, 25; and general interest members, 22. The committee is presently composed of the Advisory and 8 technical subcommittees having 15 sections and task groups.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. L. Coryell.
Vice-Chairman, W. M. Bertholf.
Secretary, R. F. Abernethy.

The Advisory Subcommittee authorized the establishment of the new Subcommittee XXIV on Statistics, with W. M. Bertholf as chairman.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of Test for:

Sulfur in Coal Ash, and
Carbon Dioxide in Coal.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Cubic Foot Weight of Crushed Bituminous Coal (**D 291 – 58 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Equilibrium Moisture of Coal at 96 to 97 per cent Relative Humidity and 30 C (**D 1412 – 56 T**).

Tentative Revision of Standard Definitions of:

Terms Relating to Coal and Coke (**D 121 – 30**).

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (**D 271 – 58**):[2]

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 8.

[2] 1958 Book of ASTM Standards, Part 8.

New Notes.—To Section 54 add the following Notes 33, 34, 35, and 36, renumbering subsequent Notes accordingly. Note 33 is to follow Paragraph (*a*), Note 34 is to follow Paragraph (*b*), Note 35 is to follow Paragraph (*c*), and Note 36 is to follow Paragraph (*f*).

NOTE 33.—The weight of coal or coke sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

NOTE 34.—Bomb parts should be inspected carefully after each use. Threads on the main closure should be checked frequently for wear. Cracked or significantly worn parts should be replaced. The bomb should be returned to the manufacturer occasionally for inspection and possibly proof firing.

NOTE 35.—The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety codes. Suitable reducing valves and adaptors for 300 to 500-psi discharge pressure are obtainable from commercial sources of compressed gas equipment.

NOTE 36.—During ignition of a sample, the operator must not permit any portion of his body to extend over the calorimeter.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Advisory Subcommittee (O. W. Rees, chairman).—In addition to its usual executive functions the Advisory Subcommittee has given attention to certain matters of policy and procedure for Committee D-5 as follows:

Instructions for USA delegates to meetings of ISO Technical Committee 27, on Solid Mineral Fuels, were prepared and approved.

The special Task Group for By-laws revision, **W. M. Bertholf**, chairman, completed a draft of proposed new "Regulations Governing Committee D-5." The Advisory Subcommittee approved this draft, with certain recommended changes, for submission to Committee D-5 for its consideration.

A revised subcommittee responsibility list was approved for submission to Committee D-5 for action. This involves responsibility assignment of all standards and tentatives under the jurisdiction of Committee D-5, as well as specifications now under development, to specified subcommittees. Furthermore, it includes the recommendation that certain currently large specifications be broken into individual specifications with individual designation numbers.

Subcommittee II on Nomenclature and Definition (C. H. Sawyer, chairman) has been formally charged with review of all tentatives as soon as they are officially adopted by Committee D-5. Several concrete suggestions regarding definitions within currently outstanding tentatives were formulated and have been transmitted to the appropriate subcommittee chairmen for consideration by the sponsor groups. A beginning was made on a review of consistency of terminology within all of the standards on coal and coke.

Subcommittee VII on Pulverizing Characteristics of Coal (H. F. Yancey, chairman) is considering revision of Method D 409. In this connection, James Jonakin presented data on 85 coals for which he had determined grindability according to the standard and according to a modification in which 16- to 100-mesh coal was used. Interest in using a wider range of sizes stems from the fact that with some fuels, such as pipeline coal, fluid coke, or anthracite silt the specified range comprises only a few per cent of the total sample and therefore is not representative. Similarly, with mechanical sample preparation the 16- to 30-

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

mesh fraction may be very small. R. M. Hardgrove of Babcock & Wilcox Co. presented information showing the results of grindability tests made on lignite at various moisture contents.

Subcommittee XV on Plasticity and Swelling of Coal (Michael Perch, chairman).—The Method of Test for Free-Swelling Index of Coal (D 720 - 57) is under study to provide electrical heating as an alternate, and to improve the sample preparation procedure so that interlaboratory variations may be reduced.

The subcommittee prepared the Gieseler method for measuring the plastic properties of coal, as noted earlier in the report. A task group is studying the constant torque modification which gives promise of improving the operation.

The first draft of the movable-wall oven for measurement of carbonization pressures of coal was prepared and reviewed. Results of a questionnaire on the design and operation of the sole-heated test oven were tabulated and reviewed preliminary to preparation of a first draft of a method.

Subcommittee XX on Sampling and Fineness Test of Pulverized Coal (E. L. Rogers, chairman) held one meeting in the past year, in New York on Feb. 18, 1960. At this meeting J. Jonakin of Combustion Engineering presented some data on comparative testing of the Combustion Engineering sampling tip with a 7-in. cyclone and with a $1\frac{3}{4}$-in. cyclone.

The subcommittee is reviewing a draft revision of Method D 197.

Subcommittee XXI on Methods of Analysis (R. F. Abernethy, chairman) completed the Method of Test for Sulfur in Coal Ash, the Method for the Determination of Carbon Dioxide in Coal, and the Safety Notes for inclusion in Methods D 271 - 58, as noted earlier in the report.

Work is progressing on revisions of present methods for determining fusibility of coal ash and volatile matter of coal by the modified method. The subcommittee has recommended that Method D 1412 - 56 T be continued as tentative. Data were submitted by cooperating laboratories for methods under consideration as possible standards for determining chlorine and forms of sulfur in coal.

The subcommittee approved a proposal that only one method for determining total moisture in coal be standardized. Work toward this end will go forward.

Subcommittee XXII on Physical Tests of Coke (W. L. Glowacki, chairman).—The work of this subcommittee was interrupted by the sudden death of the previous chairman, B. P. Mulcahy. At the first meeting under the new chairman it was decided to use the task group method in the forthcoming review of ASTM procedures on the physical tests of coke. Emphasis will be placed first on the group including Methods D 141, D 293, and D 294.

Subcommittee XXIII on Sampling (W. M. Bertholf, chairman).—Substantial progress has been made in the preparation of methods for sample preparation and mechanical sampling.

Tentative agreement was reached with Subcommittee XXI on the moisture procedure to be specified in the method for sample preparation.

Subcommittee XXVII, American Group ISO/TC 27 on Solid Mineral Fuels (W. H. Ode, chairman) is charged with the responsibility of conducting USA participation for Committee D-5 through the American Standards Assn. in the work of Technical Committee 27, on Solid Mineral Fuels, of the International Organization for Standardization. In order to expedite handling the many items involved, four Research Sections on: (1) Sampling, (2) Preparation Terminology and Performance, (3) Coke Testing, and (4) Analysis were organized within the subcommittee.

During the year 7 Draft ISO Proposals have been reviewed and consensus recommendations submitted to the ASA for submission to ISO/TC 27.

Since the last annual report was prepared the United States has been represented at ISO/TC 27 meetings as follows: Working Group 7, Sampling (London, England, May, 1959): W. W. Anderson, Leader, W. H. Ode, and G. L. Barthauer; Subcommittee 1, Coal Preparation Terminology and Performance (London, England, May 1959): W. W. Anderson, Leader, W. H. Ode, and G. L. Barthauer; Subcommittee 2, Brown Coal and Lignite (Warsaw, Poland, June 2–4, 1959): W. H. Ode; Working Group 8, Coke Testing (Rome, Italy, Nov., 1959): W. L. Glowacki, Leader, and C. R. Montgomery.

It seems pertinent to comment that the fund for support of future travel expenses of delegates is quite limited; hence future USA representation will depend on further contributions by interested groups. On the spot representation is necessary for effective participation.

Task Group C, Adiabatic Calorimeter (A. O. Blatter, chairman).—Twelve of the sixteen members have reported their results of a series of calibrations. From these data, Task Group C concluded that the water equivalent of the calorimeter should be the average of 10 determinations made over a period of not less than 3 days nor more than 5 days. Also, to be acceptable, the standard deviation of the series shall be 6.5 or less.

The next step will be performing a round-robin test on coal samples. Four different coals in duplicate will be supplied to each of the participants. Three analyses on each of the pair of pulps are to be made. These data should supply Task Group C with the information for establishing reproducibility values between laboratories and within laboratories. This work is now in progress.

This report has been submitted to letter ballot of the committee, which consists of 63 voting members; 58 members returned their ballots, of whom 55 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

O. W. REES,
Chairman.

R. L. CORYELL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-5 presented to the Society through the Administrative Committee on Standards the recommendation that the Method of Test for Plastic Properties of Coal by the Gieseler Plastometer be published as tentative. This recommendation was accepted by the Standards Committee on September 29, 1960, and the new tentative method appears in the 1960 Supplement to the Book of ASTM Standards, Part 8, bearing the designation D 1812 – 60 T.

REPORT OF COMMITTEE D-6
ON
PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 25, 1959, in Atlantic City, N. J., and on Feb. 25, 1960, in New York, N. Y. The Advisory Committee, subcommittees, and task groups held meetings in conjunction with the main committee.

The committee consists of 98 members, of whom 81 are voting members; 30 are classified as producers, 22 as consumers, and 46 as general interest members.

Committee D-6 regrets to report the death of Lewis S. Reid, past chairman of the committee. His contribution to the work of ASTM and Committee D-6 was recognized shortly before his death by his becoming a recipient of the Award of Merit.

Committee D-6 is jointly preparing with TAPPI a Symposium on Relative Humidity, Its Measurement and Control, to be held during the TAPPI Testing Conference in Grand Rapids, Mich., Sept. 26–29. This effort reflects the importance placed on this significant topic.

A task group has been appointed to revise the monograph "Paper and Paperboard: Characteristics, Nomenclature and Significance of Tests" (*ASTM STP No. 60-A*). A Glossary of Terms relating to subjects in the monograph is to be included.

The ASTM-TAPPI Joint Coordinating Committee's method of operation has been revised and approved by the committee.

The officers elected for the ensuing term of two years are as follows:

Chairman, Henry Birdsall.

Vice-Chairman, T. W. Lashof.

Secretary, R. E. Green.

Members-at-Large, Advisory Committee: C. F. Ackermann and M. L. Taylor.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Recommended Practice for Interlaboratory Evaluation of Test Methods Used with Paper and Paper Products as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Chloride Content of Paper and Paper Products (**D 1161 – 51 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Title.—Change to read "Method of Test for Total Chloride Content of Paper and Paper Products."

Section 1.—In the second line change "chloride" to "total chloride." Delete the Note under this section.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 6.

[2] 1958 Book of ASTM Standards, Part 6.

New Section.—Add a new Section 2 entitled "Significance" to read as follows, renumbering subsequent sections accordingly:

2. *Significance.*—Excessive amounts of chlorides in paper indicate the presence of electrolytes which may affect the permanence of paper in certain industrial applications. Chlorides may also cause corrosion of metal in contact with the paper in such applications as wire insulation and in packaging of polished metal articles.

NOTE.—A method for determination of water-soluble chlorides is included in ASTM Method D 202.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Tensile Breaking Strength of Paper and Paper Products (D 828 - 48):[2] Revise as appended hereto.[3]

Standard Method of Test for Ring Crush of Paperboard (D 1164 - 53):[2]

Section 1.—Divide into Sections 1 and 2 entitled "Scope" and "Significance" to read as follows, renumbering subsequent sections accordingly:

1. *Scope.*—This method covers the procedure for measuring the resistance of paperboard to edgewise compression. It is intended only for paperboard having a thickness not exceeding 0.036 in.

2. *Significance.*—Corrugated and solid fiberboard containers are subject to crushing forces in shipment. This test is used for two purposes in the evaluation of the paperboard to be used as components of such fiberboard containers: (1) to indicate edgewise rigidity of the board so that manufacturing processes may be controlled to secure the desired results, and (2) to indicate the probable crushing resistance of the finished container.

Section 2.—In Paragraph (*a*) change the speed from "15 ± 5 lb per sec" to "25 ± 5 lb per sec." In Paragraph (*c*) change the last sentence to read: "Parallelism of the long edges of the specimen shall be such that the width dimension at opposite ends shall be within 0.0006 in. of each other. The length shall be 6 ± 0.005 in."

Section 3.—Change lines 11 and 12 to read ". . . (1) the long edges are parallel such that the width at opposite ends shall be within 0.0006 in. of each other . . . ."

Section 4.—Change the reference to read "Standard Method of Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (D 641 - 49)."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sampling and Conditioning (T. W. Lashof, chairman) is working jointly with TAPPI on a study of the effects of relative humidity on the physical properties of paper. The task groups on sampling and conditioning are continuing their efforts to develop significant standard conditions and improve paper sampling.

Subcommittee II on Chemical Methods (A. P. Tracy, chairman).—A subcommittee ballot on a new method for colorimetric determination of starch content is being taken. A method on mineral fillers and coatings is being completed. A task group will study revisions of the method for sulfur determination. Methods for determining mold or mildew resistance are being developed.

---

[3] The revised standard appears in the 1960 Supplement to Book of ASTM Standards, Part 6.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Paper Testing (L. Price, chairman).—A revision of Method D 824 is ready for committee ballot. Work is progressing on review and revision of Methods D 645, D 987, D 1223, and D 1270.

Subcommittee IV on Physical Test Methods for Container Board (F. D. Long, chairman) is revising the flat crush of corrugated board and static bending test for corrugated paperboard for publication next year. A new method on column compression tests is being studied.

Subcommittee VI, Editorial (W. R. Willets, chairman).—Significance statements are being prepared for the remaining Committee D-6 standards. The subcommittee has been charged with revision of the Monograph, Paper and Paperboard; Characteristics, Nomenclature and Significance (*STP No. 60 A*).

This report has been submitted to letter ballot of the committee which consists of 98 members; 46 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. H. CARTER,
Chairman.

R. E. GREEN,
Secretary.

REPORT OF COMMITTEE D-7
ON
WOOD*

Committee D-7 on Wood and ten of its subcommittees met at the Forest Products Laboratory, Madison, Wis., on January 21 and 22, 1960. The following subcommittees met: Subcommittee I on Specifications for Timber; Subcommittee II on Laminated Timber; Subcommittee V on Methods of Preservative Treatment of Timber; Subcommittee VI on Timber Preservatives; Subcommittee VII on Wood Poles and Cross Arms; Subcommittee IX on Methods of Testing; Subcommittee XI on Moisture Content of Timber; Subcommittee XII on Fire Performance; Subcommittee XII on Durability and Exposure; and Subcommittee XV on Wood-Base Fiber and Particle Panel Materials.

A social hour and dinner meeting of Committee D-7 members and guests was held at the Towne Club on the evening of January 21. Following the dinner, an illustrated talk entitled "This is Spain!" was given by Mr. Markwardt. The talk covered details of his trip to Spain as the U. S. Delegate to the Fourth Conference on Wood Technology, under the sponsorship of the Food and Agriculture Organization of the United Nations.

Two symposiums were held at the Third Pacific Area National Meeting of the Society under the sponsorship of Committee D-7. The Symposium on Wood in Building Construction consisted of nine papers presented in two one-half day sessions; the Symposium on Treated Wood for Marine Use consisted of seven papers.[1]

The committee accepted with regret the resignation of Lyman W. Wood as Vice-Chairman.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. J. Markwardt.
Vice-Chairman, W. G. Youngquist.
Secretary, W. A. Oliver.

New Tentatives

The committee recommends for publication as tentative the following proposed methods and specifications as appended hereto:[2]

Tentative Methods of:

Testing Metal Fasteners,
Conducting Shear-Block Test for Quality Control of Glue Bonds in Scarf Joints,
Evaluating Wood Preservatives by Field Tests with Stakes, and
Chemical Analysis of Wood Charcoal.

Tentative Specification for:

Pressure Treatment of Timber Products.

Revision of Tentatives

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Test for Evaluating the Properties of Building Fiber-

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The papers presented at these symposiums are to be included in *ASTM STP No. 275* and *No. 282*.

[2] The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

boards (D 1037 - 56 T):[3] Revise as appended hereto.[2]

Tentative Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials (D 1554 - 58 T):[3]

General Definitions.—Add the following new definition: "**Wood-Cement Board.**—A panel material where wood usually in the form of excelsior is bonded with inorganic cement."

Terms Relating to Wood-Base Fiber and Particle Panel Materials.—Revise the definitions for "flakes," "shavings," and "tempering" to read as follows:

Flakes.—Specially generated thin flat particles of predetermined uniform thickness and predetermined length(s) with the grain of the wood essentially parallel to the flat surface and with dimensions usually wide and long with respect to the thickness.

Shavings.—Thin slices or strips of wood of varying thickness pared off with a knife, planer, or other cutting instrument in which the cut may be either across, parallel to, or at an angle to the axis of the fibers.

Tempering.—The manufacturing process of adding to a fiber or particle panel material a siccative material such as drying oil blends of oxidizing resin which are stabilized by baking or other heating after introduction.

Add a new definition for "heat treating" to read as follows:

Heat Treating.—The process of subjecting a wood-base panel material (usually hardboard) to a special heat treatment after hot pressing to increase some strength properties and water resistance.

Terms Describing Wood-Base Fiber and Particle Panel Products.—Add new definitions for "tempered hardboard" and "standard hardboard" to read as follows:

Tempered Hardboard.—A hardboard subjected to tempering as previously defined or specially manufactured with other variation in usual process so that the resulting product has special properties of stiffness, strength, and water-resistance associated with boards meeting specifications for that quality product.

Standard Hardboard.—Hardboard substantially as manufactured at the end of hot pressing, except for humidification to adjust moisture content, trimming to size, and other subsequent machining, and having the properties associated with hardboard meeting specifications for that quality product.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Alpha-Cellulose in Wood (**D 1103 - 55 T**),[3] and Methoxyl Groups in Wood and Related Materials (**D 1166 - 55 T**).[3]

Tentative Specifications for:

Modified Wood (**D 1324 - 57 T**).[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. W. Wood, chairman).—The principal activity during the past year has been in connection with basic stresses for the structural design of wood. Following extensive strength studies by the Forest Products Laboratory and some by the lumber industry, a number of conferences were held in an attempt to work out reasonable and just basic stresses for Douglas fir and other species. These conferences led to a thorough re-examination of the Laboratory policy with respect to the establishment of basic stresses and working stresses for wood. The Laboratory adopted the position that it will not establish basic stresses, but will continue to participate fully in the deliberations of ASTM Committee D-7, which is now responsible for published basic stresses in Methods D 245, leaving the final decision to that group, or to any other similarly qualified group. The revision of Method D 245 has been referred to a small task group.

---

[3] 1958 Book of ASTM Standards, Part 6.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A plan to establish working stresses for nonstructural grades of timber on a statistical basis from a strength survey of the grades without using basic stresses or strength ratios was proposed for inclusion in a revision of Method D 245. Included was a proposal for an increase of working stresses for these grades based on the concept of load-sharing members for light-frame construction as in joists and rafters in residential construction. The subcommittee decided that this proposal should be handled as a separate item from Method D 245, and the proponent was requested to submit them to the subcommittee in that form for consideration.

These matters will continue to occupy the attention of Subcommittee I during the coming year.

Subcommittee II on Laminated Timber (F. J. Hanrahan, chairman).—Study is being continued on the possibility of reducing the time required for the cyclic tests in Method D 1101 - 58. The time element in conducting the tests is a critical factor in the use of the standard, and a quicker cycle that would produce equally satisfactory results would be particularly helpful.

The subcommittee is also studying the problem of quality control in fabrication of glued-laminated members and the development of tests for this purpose. Under consideration are splitting tests of glued joints and tension tests for finger joints.

The subcommittee has recommended the adoption of a modified shear-block test for application to plain scarf joints as well as face joints, and reference to this item is included elsewhere in this report.

Subcommittee V on Methods of Preservative Treatment of Timber (C. W. Best, chairman).—The work of the subcommittee has culminated in the completion of a proposed Specification for Pressure Treatment of Timber Products referred to elsewhere in this report.

Subcommittee VI on Timber Preservatives (D. L. Davies, chairman).—Present specifications for wood preservatives are developed with the thought of having a separate and independent specification for each different preservative. With the considerable number of wood preservatives now employed commercially, there is an obvious repetition of procedures when two different preservatives contain a common ingredient. An argument in favor of this duplication is that it is not possible to write a common procedure for methods of analysis for all the preservatives without becoming very voluminous as to modifications to be made in specific cases. The argument in favor of condensing the standards is that it avoids repetition, as illustrated by the American Wood-Preservers' Assn. standards. Study of this question is being taken up by the subcommittee with the thought of establishing a firm policy as to the preferable procedure in preparing specifications for wood preservatives.

Subcommittee VII on Wood Poles and Cross Arms (R. P. A. Johnson, chairman).—The subcommittee met to review the status of the Wood Pole Research Program and consider plans for further work.

The drafting of the final report on the ASTM Wood Pole Research Program was completed in March, and copies have been sent for review to the Technical Advisory Committee, after which it will be forwarded to ASTM for publication and general distribution. The report summarizes, analyzes, and interprets the data from the ASTM program, together with the results of a considerable body of related research bearing on specifications for wood poles for structural use. Included in the comprehensive report are data on the strength of poles and small clear specimens cut from the pole material, the effects of preservative treatment, moisture content and shrinkage of poles, and spiral grain and twist of poles.

The subcommittee took cognizance of the fact that the present program did not answer all the questions on poles and further research is needed in the fields of pole selection by specific gravity, effects of hand versus machine peeling, and butt versus full-length soaking. The present importance of poles for building framing was recognized, and it was agreed that working stresses for this use are within the scope of work of ASTM Committee D-7. Reports were heard on proposed revisions of the American Wood-Preservers' Assn. treating specifications and on the activities of American Standards Assn. Committee 05 on Wood Poles. The subcommittee will consider these and other matters relating to wood poles and cross arms during the coming year.

Subcommittee VIII on Modified Wood and Wood-Base Materials (W. G. Youngquist, chairman).—A tentative specification for modified wood employing wood in the form of lumber or veneer or an assembly of lumber and veneer has been in use for several years. No revisions have been required in this method, and it was accordingly recommended that Specifications D 1324 be adopted as standard.

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman).—Over a period of years, a number of test methods for various metal fasteners and fastening devices employed with wood have been developed and extensively used but have not been written up or formalized as standards. The principal activity of the subcommittee during the year was the development of procedures in the metal fastening field covering a screw withdrawal test method, a lateral nail or screw resistance test method, and a method of testing timber connector joints. These have been included in proposed new tentatives as noted elsewhere in this report.

Further work during the coming year includes consideration of developing methods of testing strap hangers for joists, methods of tests for flooring, methods of determining specific gravity, including small sample techniques, methods of testing plywood in large-size elements, and statistical methods of sampling for tests of small clear specimens. The subcommittee also is reviewing the possible need of developing definitions relating to mechanical fasteners.

Subcommittee XI on Moisture Content of Timber (R. C. Rietz, chairman).—At a meeting of the subcommittee, developments in moisture content testing equipment during the past year and the status of information on the applicability of nuclear emission methods to wood moisture content determination were reported on and discussed. The development of a practical method of moisture content determination using radioactive isotopes does not appear encouraging because of the influence of variable specific gravity.

A task group within the subcommittee was organized to prepare portions of the proposed tentative standard for wood moisture determination. The new tentative will include the oven drying method, distillation methods, electrical methods, and hygrometric methods.

Subcommittee XII on Fire Performance (H. O. Fleischer, chairman).—The work of this subcommittee is being reactivated and a well-attended subcommittee meeting was held. The major part of the meeting was devoted to a reappraisal of the field of work of the subcommittee, with general discussion on fire problems in the use of wood and wood products. It was agreed that the subcommittee could perform a very useful function by adopt-ing an aggressive program of studying fire problems relating to these materials. A considerable number of problems were discussed, indicating fruitful fields of study. On the basis of this discussion, it was agreed to broaden the membership of the subcommittee and to appoint task groups as needed to carry on the work.

Because of the broadened field of work contemplated, it was recommended that the name of the subcommittee be changed from "Subcommittee XII on Fire-Retardant Wood" to "Subcommittee XII on Fire Performance."

The members of the subcommittee witnessed tests in the Forest Products Laboratory's new thermogravimetric balance and also in the 8-ft tunnel apparatus for flame-spread tests.

Subcommittee XIII on Durability and Exposure (R. M. Lindgren, chairman).—The subcommittee has had an active program during the year and held two well-attended meetings.

The chairman reported that a slightly modified version of Method D 1413 had recently been adopted by the National Woodwork Manufacturers' Assn., in place of a previously used method. This action by NWMA represented another example of the widespread recognition of the value of Method D 1413 as an accelerated test. Discussion of this tentative standard at the Madison meetings centered around the adequacy of the present weathering procedure, particularly for volatile preservatives such as coal-tar creosote. It was recognized by the subcommittee that the eventual inclusion of several types of weathering, depending on the kind of preservative to be tested, might be necessary. It was recommended that this method be continued as tentative without change.

A principal accomplishment was the development of a new procedure entitled Tentative Method of Evaluating Wood Preservatives by Field Tests with Stakes, as noted earlier in the report.

Ways and means are being considered to study a procedure for evaluating wood piles in sea water, subject of a paper presented at the Third Pacific Area Meeting.

The work of the subcommittee in developing accelerated laboratory and field tests of preservatives was commended in a recent letter from the Prevention of Deterioration Center, National Research Council.

Subcommittee XIV on Methods of Chemical Analysis (W. E. Moore, chairman).—The subcommittee reviewed the various standards under its jurisdiction and recommended the adoption as standard of Methods D 1103 and D 1166.

The subcommittee has completed a proposed Tentative Method of Chemical Analysis of Wood Charcoal, as noted earlier in the report, based on extensive study and developmental work at the Forest Products Laboratory in connection with the analysis of charcoal samples obtained in kiln research work, following clearance with Committee D-5 on Coal and Coke.

Subcommittee XV on Wood-Base Fiber and Particle Panel Materials (W. C. Lewis, chairman).—This subcommittee has a very active program of work and has held three well-attended meetings.

Some definitions were revised and other new definitions were accepted, as noted elsewhere in this report, in Definitions D 1554.

Some revisions to Methods D 1037 were approved by the subcommittee, as noted elsewhere in this report. Some new test methods relating to requirements of hardboard specifications were approved, as well as procedures for screw withdrawal, abrasion, and falling ball impact.

The Task Force on Insulating Roof Deck Slab is considering test procedures developed around work at the Forest Products Laboratory and the Insulation Board Institute for determining structural properties. This work is also current in ASTM Committee C-16 on Thermal Insulating Materials. However, jurisdiction on this procedure has been given to Committee D-7, and the new tentative is recommended for acceptance elsewhere in this report.

A Task Force on High-Density Insulating Board has been appointed and has listed the properties important to the proper use of the material. It is surveying ASTM procedures for measuring these properties and is correlating its activities with such groups as the Insulation Board Institute. The Task Force on Makeup, Direction, and Need for Specifications for Particle Board has agreed that some activity in this field is warranted in view of the growth of the industry, and will continue its activity.

This report has been submitted to letter ballot of the committee, which consists of 104 members; 60 members returned their ballots, of whom 57 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

W. A. OLIVER,
Secretary.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS MATERIALS FOR ROOFING, WATERPROOFING, AND RELATED BUILDING OR INDUSTRIAL USES*

Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses held two meetings during the year: in Atlantic City, N. J., on June 23, 1959, and in Chicago, Ill., on Feb. 3, 1960.

The membership consists of 102 voting members, of whom 68 are classified as producers, 10 as consumers, and 24 as general interest members.

The committee suffered the loss through resignation of T. G. Hayes of Owens Corning Fiberglas; J. E. Boarini of Celotex Corp.; H. A. Pasman of the Erie Railroad; and J. Weisberg of Frankford Arsenal, U. S. Army.

A new subcommittee has been formed to develop standards for industrial pitches.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. R. Snoke.
Vice-Chairman, M. R. Beasley.
Secretary, H. B. Keene.

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of the Standard Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (**D 313 - 41**):[1]

Section 1.—Change to read as follows:

1. *Scope.*—This method of test covers a procedure for determining that portion of the mineral or other insoluble matter in asphaltic mixtures which is retained on the No. 200 (74-micron) sieve.

REVERSION OF STANDARD TO TENTATIVE

The committee recommends reversion of the Standard Method of Test for Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (**D 452 - 40**)[1] to tentative.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions as indicated of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (**D 173 - 44**)[1] (Tentative Revision issued June, 1957),

Asphalt Shingles Surfaced with Mineral Granules (**D 225 - 58**)[1] (Tentative Revision issued June 1958),

Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-up Roofs (**D 226 - 56**)[1] (Tentative Revision issued June 1957),

Asphalt Roll Roofing Surfaced with Mineral Granules (**D 249 - 56**)[1] (Tentative Revision issued June 1958), and

Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (**D 250 - 56**)[1] (Tentative Revisions issued June 1957, June 1958, and June 1959).

Standard Method of Test for:

Coarse Particles in Mixtures of Asphalt and Mineral Matter (**D 313 - 41**)[1] (Tentative Revisions issued June 1958).

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Part 4.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets, and Shingles (**D 228 – 57 T**).[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 102 members; 64 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. R. SNOKE,
Chairman.

G. W. ROBBINS,
Secretary.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS*

Committee D-9 on Electrical Insulating Materials held three meetings during the year: in Atlantic City, N. J., June 23–25, 1959, during the Annual Meeting of the Society; in Cleveland, Ohio, Sept. 21–23, 1959; and in Cincinnati, Ohio, March 9–11, 1960.

At the time of the Cincinnati meeting, the membership comprised 212, of whom 96 were classified as producers, 67 as consumers, and 49 as general interest members.

Organizational changes during the year included completion of the organization of the new Subcommittee XIII on Composite Insulating Materials. The D-9 Advisory Committee also authorized the formation of the new Subcommittee XVI on Thermal Endurance, and of a new subcommittee on dielectric films, both of which are still to be organized. A survey of interest in the setting up of West Coast sections of D-9 subcommittees was authorized. Revisions of the regulations governing Committee D-9 have been approved by committee letter ballot.

Committee D-9 has continued to work with other groups concerned with electrical insulation, including the International Electrotechnical Commission (IEC), the International Organization for Standardization (ISO), the American Institute of Electrical Engineers (AIEE), and the American Standards Assn. (ASA). The committee has assisted in directing a research project at the University of Delaware on insulation resistance testing of printed circuit boards, sponsored jointly by the National Electrical Manufacturers Assn. (NEMA), the Electronic Industries Assn. (EIA), and ASTM. E. B. Curdts was appointed as the ASTM representative to the AIEE Electrical Insulation Committee. Kenneth Wechsler and Harold S. Endicott were appointed to represent ASTM on the new ASA Sectional Committee on Insulation Coordination (C 92) which is sponsored jointly by AIEE and NEMA.

The committee held a panel discussion on Determination of Cure and Useful Properties of Electrical Insulating Varnishes, at the September, 1959, meeting. A Symposium on Tracking Tests was held at the March, 1960, meeting. In October, 1959, the committee sponsored a Symposium on Electrical Insulating Materials at the Third Pacific Area National Meeting of the Society in San Francisco. The committee also sponsored a session on Dielectric Strength at the Second National Conference on the Application of Electrical Insulation, Washington, D. C., December, 1959.

The officers elected for the ensuing term of two years are as follows:

Chairman, A. H. Scott.
Vice-Chairman, E. B. Snyder.
Membership Secretary, J. R. Taylor.
Recording Secretary, Thomas Hazen.

AMERICAN STANDARDS

The American Standards Association, upon recommendation of Sectional Committee C 59 on Electrical Insulating Materials, approved as American Standard the Standard Methods of Test for Electrical Resistance of Insulating Materials (D 257 - 58; ASA C 59.3 - 1959).

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the recommendations listed below. These recommendations were accepted by the Standards Committee on the dates indicated.

New Tentative Definitions of:

Electrical Terms (**D 1711 - 60 T**) (Approved March 23, 1960).

Revision of Tentative Methods of Test for:

A-C Capacitance, Dielectric Constant, and Loss Characteristics of Electrical Insulating Materials (**D 150 - 59 T**) (Approved Dec. 28, 1959).

Revision of Tentative Methods of Testing:

Nonrigid Vinyl Chloride Polymer Tubing (**D 876 - 59 T**) (Approved Nov. 23, 1959), and

Fully Cured Silicone Rubber Coated Glass Fabric and Tapes for Electrical Insulation (**D 1458 - 59 T**) (Approved Dec. 28, 1959).

Tentative Revision of Standard Methods of Test for:

Dielectric Breakdown Voltage and Dielectric Strength of Electrical Insulating Materials (**D 149 - 59**) (Approved Nov. 23, 1959), and

Electrical Resistance of Insulating Materials (**D 257 - 58**) (Approved Nov. 23, 1959).

Tentative Revision of Standard Methods of:

Testing Sheet and Plate Materials Used for Electrical Insulation (**D 229 - 58**) (Approved Nov. 23, 1959).

Tentative Revision of Standard Specifications for:

Communication and Signal Pin-Type Lime-Glass Insulators (**D 879 - 58**) (Approved Nov. 23, 1959).

Revision and Reversion to Tentative of Standard Methods of:

Testing Vitrified Ceramic Materials for Electrical Applications (**D 116 - 59 T**) (Approved Nov. 23. 1959).

The new tentative Definitions D 1711 will appear in the 1960 Supplement to Book of ASTM Standards, Part 9; the other recommendations appear in the 1959 Supplement to Book of ASTM Standards, Part 9.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation (**D 202 - 55 T**):[1]

Section 3.—Add a new Paragraph (*c*) to read as follows:

> (*c*) For work of such precision that the hysteresis in the equilibrium moisture content may lead to an appreciable error, the moisture content equilibrium under standard conditions shall be approached from a drier state by first reducing, if necessary, the moisture content to less than half the value under standard conditions, and then conditioning under standard conditions. For that purpose, the specimens may be dried in a desiccator or by other convenient means, provided the temperature does not exceed 60 C (140 F).

Tentative Methods of Testing Nonrigid Vinyl Chloride Polymer Tubing (D 876 - 59 T):[2] Revise as appended hereto.[3]

Tentative Methods of Testing Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (**D 1000 - 59 T**):[2]

Sections 39 to 41.—Delete these sections, which cover the Storage Stability (Accelerated Heat and Moisture Test) Method, renumbering subsequent sections accordingly.

Section 57.—In the second (and final) sentence of Paragraph (*a*), replace the phrase "for 90 min" with the phrase "for a predetermined time," and remove the phrase "whichever occurs first."

---

[1] 1958 Book of ASTM Standards, Part 9.

[2] 1959 Supplement to Book of ASTM Standards, Part 9.

[3] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 9.

In the fourth (and final) sentence of Paragraph (*b*), replace the phrase "for 30 min" with the phrase "for a predetermined time," and remove the phrase "whichever occurs first."

Section 58.—In Item (4), remove the phrase "or statement of no failure."

Tentative Methods for Sampling and Testing Untreated Mica Paper Used for Electrical Insulation (D 1677 - 59 T):[2]

Section 17.—Change the next to last sentence to read: "Tests shall be made in air using 2-in. electrodes and the short-time (continuous rise) method."

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Methods of Testing Molded Materials Used for Electrical Insulation (**D 48 - 54 T**)[1] because these methods no longer serve a useful purpose. Steps have been taken to have all references to Methods D 48 removed from specifications prepared by Committees D-9 and D-20.

TENTATIVES CONTINUED WITHOUT REVISION

The committee has reviewed all other tentatives under its jurisdiction which have been published for two years or longer without revision and recommends their continuation without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (W. E. Harvey, chairman) sponsored the panel discussion on cure and useful properties of electrical insulating varnishes referred to earlier in this report. The subcommittee is working on a revision of Tentative Method of Test for Set Time of Thermosetting Phenol-Formaldehyde Varnishes (D 1472 - 57 T). Work is also in progress on revision of Standard Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 55) in order to have it conform to Military Standard MIL-V-1137A. The subcommittee is engaged in several investigations including use of the Brookfield viscometer, deterioration of varnishes at elevated temperatures, and "fluidized bed" materials.

Subcommittee II on Effects of Nuclear and High Energy Radiation (D. J. Metz, chairman).—This joint D-9/D-20 subcommittee is revising the Tentative Recommended Practice for Exposure of Polymeric Materials to High Energy Radiation (D 1672 - 59 T). During the year close liaison with Committee D-11 on Rubber and Rubber-like Materials was established. Some reorganization of the subcommittee has been accomplished and an effort is being made to promote close coordination with the work of Committee E-10 on Radioisotopes and Radiation Effects.

Subcommittee III on Plates, Tubes, Rods, and Molded Materials (A. J. Balch, chairman).—Mr. Balch resigned from the chairmanship of this subcommittee prior to the March, 1960, meeting and K. Wechsler became acting chairman. At the March, 1960, meeting the subcommittee submitted for D-9 letter ballot a complete revision of Methods D 229, with the recommendation of reversion to tentative. Revisions of the joint D-9/D-20 Tentative Specifications for Laminated Thermosetting Materials (D 709 - 55 T) have been approved by subcommittee letter ballot and forwarded to Subcommittee XVIII of Committee D-20. The subcommittee initiated the recommendation for withdrawal of Methods D 48 as noted earlier in this report. The subcommittee has recommended retention as tentative of Specifications D 709, D 710, D 1202, D 1532, and D 1592 because of revisions under way or contemplated. Work is continuing on a proposed tentative specification for copper-clad laminates. A task group of the section on electrical properties has completed interlaboratory tests on insulation resistance of copper-clad laminates.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee V on Ceramic Products (A. H. Scott, chairman) sponsored the revision and reversion to tentative of Methods D 116 and the tentative revision of Specifications D 879 accepted by the Standards Committee. The subcommittee has recommended for committee letter ballot the removal of the test for effect of heat on flexural strength from Methods of Testing Vitrified Ceramic Materials for Electrical Applications (D 116 – 59 T). The subcommittee is balloting a revised draft of a proposed method of test for electrical resistance of insulating materials at elevated temperatures and is working on a method for measuring dielectric constant and loss at microwave frequencies in preparation for interlaboratory testing. At the March, 1960, meeting the subcommittee made suggestions to Subcommittee VI of Committee C-21 on Ceramic Whitewares and Related Products with regard to revisions of a proposed test method for determining the electrical resistance of conductive tile.

Subcommittee VI on Solid Filling, Treating, Encapsulating, and Embedding Compounds (Thomas Hazen, chairman) at the March, 1960, meeting, submitted for committee letter ballot the recommendation for advancement to standard of Tentative Methods of Testing Hydrocarbon Waxes Used for Electrical Insulation (D 1168 – 58 T), with an editorial change to substitute the new test for color D 1500 in place of Method D 155, which is being withdrawn as an ASTM tentative method as of July 1, 1960. The section on polymerizable embedding compounds of this subcommittee is working on dielectric strength and other electrical test methods for eventual inclusion in Tentative Methods of Testing Polymerizable Embedding Compounds Used for Electrical Insulation (D 1674 – 59 T).

Subcommittee VII on Flexible Sheet, Tape, and Tubing (F. P. West, chairman) sponsored the revisions of Methods D 876 and D 1458 which were accepted by the Standards Committee. The subcommittee also prepared the revision of Methods D 1000 listed earlier. At the March, 1960, meeting the subcommittee recommended continuation of Methods D 350 and Specifications D 372 and D 373 as tentative because of revisions currently in progress and obtained Committee D-9 approval to withdraw the Suggested Method for Evaluation of Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes: Dielectric Prooftest Method, which appears as Appendix III in the October, 1959, compilation of ASTM Standards on Electrical Insulating Materials.

Subcommittee VIII on Insulating Papers (P. L. Staats, chairman) sponsored the revision of Methods D 202 listed above. At the March, 1960, meeting the subcommittee submitted for D-9 letter ballot revisions of Tentative Specifications for Electrical Insulating Paper—Interlayer Type (D 1305 – 54 T); revisions of Methods D 202 to change the thickness method and add methods for dry dissipation factor and surface friction of paper were also submitted. The subcommittee has also recommended continuation of Tentative Specifications for Absorbent Laminating Paper for Electrical Insulation D 1080 – 58 T) as tentative pending further study.

Subcommittee IX on Mica Products (K. G. Coutlee, chairman) sponsored the revision of Methods D 1677 noted above. At the March, 1960, meeting the subcommittee submitted to simultaneous letter ballot of Subcommittee IX and Committee D-9 a revision of Tentative Specifications for Natural Muscovite Mica Based on Visual Quality (D 351 - 57 T) to make the requirements for Stained A Quality mica less severe and restrictive. In its capacity as U.S.A. Committee for ISO 56 on Mica, working through ASA, the subcommittee has engaged in reviewing documents relating to visual classification of muscovite mica and methods of grading phlogopite mica blocks, thins, and splittings by size. A paper on the Armcorp Waviness Tester, including test results, is being prepared for presentation at the Subcommittee IX meeting at the 1960 Annual Meeting and for later publication in the ASTM BULLETIN. A section of the subcommittee has developed a revised method for determining the hot compression of mica plate with a proposal that it be incorporated in a revision of D 352 - 59, Methods of Testing Pasted Mica Used in Electrical Insulation.

Subcommittee X on Magnet Wire Insulation (K. N. Mathes, chairman) has effected a reorganization of its sections along applications lines. The new sections cover general purpose, high-temperature (above 180 C), and refrigerant applications, respectively. Work on thermal aging and inspection tests of magnet wire insulation is in progress.

Subcommittee XI on Nomenclature, Significance, and Statistics (C. L. Craig, chairman) prepared the new Definitions D 1711 accepted by the Standards Committee. Additional definitions of terms are in preparation. The subcommittee has continued to function in the review of all new and revised test methods and specifications to ensure conformity with proper ASTM format and usage.

Subcommittee XII on Electrical Tests (N. W. Edgerton, chairman) sponsored the revisions of Methods D 149, D 150, and D 257 accepted by the Standards Committee. At the March, 1960, meeting the subcommittee obtained D-9 approval to submit a new Suggested Test Method for Resistance to Corona Attack of Solid Electrical Insulating Materials to the editorial board for review and approval for publication in the next issue of the D-9 compilation; also approval of a request to ASTM Headquarters to provide separate reprints of the Suggested Method of Corona Measurement, which through a misunderstanding was omitted from the 1959 D-9 compilation. The subcommittee has recommended continuation of Methods D 495 and D 1389 as tentative pending investigation of the usage and results obtained by these methods for arc resistance and dielectric proof-voltage testing, respectively. Two symposia, one on dielectric strength and one on tracking tests, were sponsored by Subcommittee XII during the year, as indicated earlier in this report. At the September, 1959, meeting, the subcommittee recommended omission of the Suggested Method of Test for Dust and Fog Tracking Resistance of Electrical Insulating Materials from the 1959 D-9 compilation, with provision that copies of the method and appended comments would be made available from ASTM Headquarters on special order.

Subcommittee XIII on Composite Insulating Materials (L. J. Timm, chairman) was organized at the Annual Meeting in June, 1959. The following scope statement was approved by the D-9 Advisory Committee at the March, 1960, meeting:

> "The formulation of specifications and the preparation of methods of test for nonrigid materials combined by adhering two or more essentially integral insulating materials or

an insulating material and metallic foil to form a composite product."

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman) has in preparation a tentative revision of Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 58) in which additional standard test temperatures, higher than those now recommended, are proposed for conditioning or testing higher temperature resistant plastics and insulating materials. The work of this subcommittee is a joint activity with Committee D-20.

Subcommittee XV on Research and Technical Papers (H. S. Endicott, chairman) was responsible for the scheduling of the various symposia sponsored by Committee D-9 during the year, as mentioned earlier in this report. The chairman solicited the papers for presentation at the D-9 sponsored symposium at the Third Pacific Area National Meeting of the Society in San Francisco, October, 1959, and presided at the symposium. The subcommittee assisted in setting up the joint NEMA-EIA-ASTM research project on insulation resistance testing of printed circuit boards at the University of Delaware, and has been instrumental in distributing progress reports of this project.

This report has been submitted to letter ballot of the committee, which consists of 215 members; 157 members returned their ballots, of whom 135 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. K. GRAVES,
Chairman.

THOMAS HAZEN,
Recording Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Methods of Testing:

Vitrified Ceramic Materials for Electrical Applications (D 116 – 59 T).

Revision of Tentative Methods of Sampling and Testing:

Untreated Paper Used for Electrical Insulation (D 202 – 55 T).

Revision of Tentative Specifications for:

Electrical Insulating Paper—Interlayer Type (D 1305 – 54 T), and
Natural Muscovite Mica Based on Visual Quality (D 351 – 57 T).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the revised tentative methods and specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

REPORT OF COMMITTEE D-10
ON
SHIPPING CONTAINERS*

Committee D-10 on Shipping Containers held two meetings during the year: in East Lansing, Mich., on Oct. 14 and 15, 1959, and in Atlantic City, N. J., on April 4 and 5, 1960.

The committee consists of 96 members, of whom 34 are classified as consumers, 32 as producers, and 30 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, K. W. Kruger.
Vice-Chairman, J. W. Goff.
Secretary, R. F. Uncles.
Advisory Committee Members-at-Large: W. J. Armstrong and J. G. Turk.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision and Reversion to Tentative of Standard Methods of Test for:

Shipping Containers in Revolving Hexagonal Drum (**D 782 - 47**), and
Water Vapor Permeability of Shipping Containers (**D 1008 - 51**).

The recommendation for Method D 1008 was accepted by the Standards Committee on December 28, 1959, and that for Method D 782 on March 23, 1960. The revised methods will appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of all tentatives under its jurisdiction.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definition of Terms (C. S. Macnair, chairman) will be guided in its tabulation of terms to be added to Definitions D 996 by liaison members from each of the other subcommittees.

Subcommittee II on Methods Appraisal (C. R. Gustafson, chairman), which provides a mechanism for consideration of new test methods, has assigned the development of a test method for tapes and a method for measurement of antiskid properties to Subcommittee X, and has a number of other potential areas of investigation under study.

Subcommittee III on Moisture and Water Vapor Resistance (H. A. Bergstrom, chairman) completed its revision of the Methods of Test for Water Vapor Permeability of Shipping Containers (D 1008), and will undertake a study of the Methods of Test for Water Vapor Permeability of Packages (D 895).

Subcommittee V on Revolving Drum (M. J. Clark, chairman) is pursuing its investigation of the use of the revolving drum for purposes of ranking containers.

Subcommittee VI on Interior Packing

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

(K. Q. Kellicutt, chairman) has essentially completed its work on a compatibility test and has initiated programs covering general dynamic testing and a procedure for measuring creep. A Task Group on Instrumentation has been formed to establish correct procedures for obtaining valid data.

Subcommittee VII on Stacking Test (K. R. Martin, chairman) is preparing a Standard Practice on Dead Load Storage Life of Corrugated and Solid Fibreboard Boxes.

Subcommittee VIII on Vibration Test (A. M. Bak, chairman) has initiated a study of resonance and its effects on items in transit.

Subcommittee IX on Incline Impact Test (Guy Clark, chairman) continued its general study of this method, laying the groundwork for the preparation of test procedures. R. N. Maxson was appointed chairman of this subcommittee following Mr. Clark's resignation.

Subcommittee X on Exterior Packaging (H. W. Maas, chairman) was established by action of the Advisory Committee on April 5, 1960. The initial work of this subcommittee will be directed toward the development of test methods for packaging tapes.

This report has been submitted to letter ballot of the committee, which consists of 98 members; 93 members returned their ballots, of whom 90 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

K. W. KRUGER,
Chairman.

R. F. UNCLES,
Secretary.

REPORT OF COMMITTEE D-11

ON

RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials and its subcommittees met twice during the year: in Atlantic City, N. J. on June 26, 1959 and in Chicago, Ill. on Feb. 5, 1960.

Committee D-11 sponsored the meeting of ISO/TC 45 on Rubber at the Hudson Hotel in New York, N. Y., Oct. 26 through 31, 1959. This was a very successful meeting. ASTM and Committee D-11 are represented on all of the working groups of ISO/TC 45 through members of the American Group. The United States has accepted the convenorship of two working groups. Mr. B. S. Garvey, Jr., will be chairman of Working Group C on Unvulcanized Rubber. Mr. Rolla Taylor will be chairman of Working Group D on Physical Properties.

Committee D-11, cooperating with the Rubber Division of the American Chemical Society, and the Rubber and Plastics Division of the American Society of Mechanical Engineers, sponsored the International Rubber Conference in Washington, D. C., Nov. 8 through 13, 1959. Mr. A. E. Juve, General Chairman of the conference, reported that the cooperation of Committee D-11 added appreciably to the success of the meeting.

The following two technical papers were presented at a joint session of Committees D-11 and D-20 on Plastics on June 25, 1959: "Improved Oven Design for Accelerated Aging of Polyvinyl Chloride," by Markus Royen,[1] and "The Effect of Die Closure Pressure on Mooney Viscosity Measurement" by Norbert Mueller.

The committee was able to contribute to the financing of the ISO/TC 45 meeting and also to the International Rubber Conference. The latter operated at a small profit so that the money advanced plus 15 per cent of the profit was returned to the Committee D-11 fund. The financial report to date shows $599.46 in the fund.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. Collier.
Vice-Chairman, H. G. Bimmerman.
Secretary, J. J. Allen.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentatives:

Specifications for Low-Voltage Rubber Insulating Gloves (D 1700 - 59 T), and

Method of Test for Viscosity and Curing Characteristics of Rubber by the Shearing Disk Viscometer (D 1646 - 59 T), which represents a revision and consolidation of Methods D 927 and D 1077.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Published in the ASTM BULLETIN, No. 243, Jan., 1960, p. 43.

Revision of Tentatives:

Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 – 58 T),
Specifications for Rubber Insulating Gloves (D 120 – 52 T),
Methods of Testing Hard Rubber Products (D 530 – 57 T),
Specification for Ozone-Resisting Insulation for Wire and Cable (D 574 – 54 T),
Methods of Testing Asphalt Composition Battery Containers (D 639 – 57 T),
Specification for Elastomer Compounds for Automotive Applications (D 735 – 59 T),
Methods of Testing Rubber-Coated Fabrics (D 751 – 58 T),
Specifications and Methods of Test for Latex Foam Rubbers (D 1055 – 58a T),
Specifications and Methods of Test for Sponge and Expanded Cellular Rubber Products (D 1056 – 58 T),
Specifications for Ozone-Resisting Butyl Rubber Insulation for Wire and Cable (D 1352 – 54 T),
Recommended Practice for Description of Types of Styrene-Butadiene Rubbers (D 1419 – 59 T),
Specifications and Methods of Test for Flexible Urethane Foam (D 1564 – 58 T), and
Specifications and Methods of Test for Flexible Foams Made from Polymers or Copolymers of Vinyl Chloride (D 1565 – 58 T).

Withdrawal of Tentative Methods of Test for:

Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (D 927 – 57 T), and
Cure Characteristics of Vulcanizable Rubber Mixtures During Heating by the Shearing Disk Viscometer (D 1077 – 55 T).

These recommendations were accepted by the Standards Committee on November 23, 1959, with the exception of Methods D 639 which was accepted on March 3, 1960. The new and revised tentatives appear in the March, 1960, compilation of "ASTM Standards on Rubber Products"; all except Methods D 639 appear in the 1959 Supplement to Book of ASTM Standards, Part 9; Method D 639 will appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods of test as appended hereto:[2]

Tentative Specifications for:

Latex Dipped Goods and Coatings for Automotive Applications, and
Carbon Blacks Used in Rubber Products (Jointly with Committee D-24).

Tentative Method of Test for:

Solubility of Organic Chemicals.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods for Chemical Analysis of Rubber Products (D 297 – 59 T):[3]

These methods have been revised to combine the Tentative Methods of Identification and Quantitative Analysis of Synthetic Elastomers (D 833 – 46 T) with it into a single tentative. The revised methods are appended hereto.[2] Action approving this revision will automatically render Methods D 833 obsolete.

Tentative Specifications for Elastomer Compounds for Automotive Applications (D 735 – 59 T):[3]

Table VI.—The SAE-ASTM Technical Committee on Automotive Rubber recommends that Table VI, Physical Requirements of Type T Compound, Class TB, Heat Resistant, be completely revised as shown in the accompanying Table I.[4]

---

[2] The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

[3] 1959 Supplement to Book of ASTM Standards, Part 9.

[4] The revised table has not been included in this report but appears in the revised Specification D 735; see 1960 Supplement to Book of ASTM Standards, Part 9.

Tentative Methods of Testing Coated Fabrics (D 751 – 59 T):[3]

New Sections.—Add new Sections 41 to 45 covering a method for "Tack-Tear Resistance" to read as follows:

41. *Tack-Tear Resistance.*—This method covers a procedure for measuring the resistance of a coated fabric to tearing under considitions simulating an installation which has been tacked in place. It is intended primarily for testing vinyl plastic coated fabrics for furniture or automotive applications.

42. *Apparatus.*—The apparatus shall consist of the device illustrated in Fig. 4 (the accompanying Fig. 1)[5] and a testing machine conforming to the Specifications for Textile Testing Machines (ASTM Designation: D 76).

43. *Test Specimens.*—The specimens shall be 2 in. in width and 6 in. in length. Two sets of five specimens each are required, one set for warp tack-tear strength, having the longer dimension parallel to the filling yarns, and the other set for filling tack-tear strength having the longer dimension parallel to the warp yarn.

44. *Procedure.*—Clamp the device shown in Fig. 4 (the accompanying Fig. 1)[5] in the upper jaw of the testing machine and adjust the machine to compensate for its weight. The specimen shall be impinged on the needles, taking care that the line of needles is parallel to the threads to be tested and exactly ½ in. from the top edge of the specimen. Clamp the specimen in the lower clamp. Operate the machine as for a tension test with the distance from the bottom of the needles to the top of the lower clamp 3 in., and the rate of jaw separation 12 in. per min with no load. The faces of the bottom clamp shall measure 1 by 3 in. or more. If the machine is of the pendulum type, the pawls on the pendulum shall be disengaged from the ratchet. Record the average load necessary to tear the fabric, preferably with an autographic recording device.

45. *Report.*—Report the median of the results of the five individual tests on the warp as the warp tack-tear strength, and the median of the five individual tests on the filling as the filling tack-tear strength.

New Figure.—To the new Section 42 add a new Fig. 4 as shown in the accompanying Fig. 1.[5]

Tentative Method of Test for Accelerated Ozone Cracking of Vulcanized Rubber (D 1149 – 55 T):[6]

This method has been completely revised as appended hereto.[2]

Tentative Recommended Practice for Description of Types of Styrene-Butadiene Rubbers (SBR) and Butadiene Rubbers (BR) (D 1419 – 60 T):[7]

Table II.—Revise the column heading "Carbon Black, per cent," to read "Carbon Black, PHR," to be consistent with the manner in which "oil" is expressed. Revise values of several types as shown in the accompanying Table III.

TABLE III.—REVISIONS OF TABLE II, SPECIFICATIONS D 1419.

Type	Carbon Black, PHR	Type	Carbon Black, PHR
1100	50	1604	60
1103	50	1605	50
1104	50	1800	82.5
1600	50	1801	50
1601	50	1802	82.5
1602	50	1803	50
1603	50	1805	75
		1812	50

Tentative Methods for Testing Carbon Blacks in Rubber (D 1522 – 58 T):[6]

This method has been completely rewritten. It provides only one basic formulation using natural rubber and eliminates the formula for SBR rubber. The proposed revision is appended hereto.[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

---

[5] The new figure is not shown in this report but appears in the revised methods D 751, see 1960 Supplement to Book of ASTM Standards, Part 9.

[6] 1958 Book of ASTM Standards, Part 9.

[7] 1960 Compilation of ASTM Standards on Rubber Products.

Tentative Specifications for:

Insulated Wire and Cable: Vinyl Chloride Plastic Insulating Compound (**D 734 - 50 T**),
Heavy Duty Black Neoprene Sheath for Wire and Cable (**D 752 - 57 T**),
General Purpose Neoprene Sheath for Wire and Cable (**D 753 - 57 T**),
Ozone Resisting Butyl Rubber Insulation for Wire and Cable (**D 1352 - 59 T**), with the following editorial change: *Section 1(b)*.—Add to the end of the section the phrase "up to 28,000 v phase to phase."
Synthetic Rubber Heat or Moisture-Resisting Insulation for Wire or Cable (**D 1520 - 58 T**),
Synthetic Rubber Performance, Moisture-Resisting Insulation for Wire and Cable (**D 1521 - 58 T**), and
Synthetic Rubber Insulation for Wire and Cable, 90 C Operation (**D 1523 - 58 T**).

Tentative Methods of:

Testing Flat Rubber Belting (**D 378 - 51 T**),
Test for Resistance to Surface Cracking of Stretched Rubber Compounds (**D 518 - 57 T**),
Testing Automotive Air Brake and Vacuum Brake Hose (**D 622 - 57 T**),
Test for Volume Resistivity of Electrically Conductive Rubber and Rubber-like Materials (**D 991 - 48 T**),
Test for Evaluating Pressure Sealing Properties of Rubber and Rubber-like Materials (**D 1081 - 58 T**),
Test for Evaluating Low-Temperature Characteristics of Rubber and Rubber-Like Materials by a Temperature-Retraction Procedure (TR Test) (**D 1329 - 58 T**), and
Test for Change in Length of an Elastomeric Vulcanizate Resulting from Immersion in a Liquid (**D 1460 - 57 T**).

Tentative Recommended Practice for:

Standard Test Temperatures for Rubber and Rubber-like Materials (**D 1349 - 58 T**).

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Specifications for Natural Rubber AO Insulation for Wire and Cable, 60 C Operation (**D 27 - 58**).[6] Subcommittee V reports that this is no longer used.

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Methods of Identification and Quantitative Analysis of Synthetic Elastomers (**D 833- 46 T**).[6] These methods are being combined with Methods D 297, Chemical Analysis of Rubber.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the remain-

TABLE IV.—SBR POLYMERS TO BE ADDED TO TABLE II, SPECIFICATIONS D 1419.

Type	1510	1606	1607	1806	1807	1808
Nominal Temperature, deg Fahr	43	43	43	43	43	43
Activator	FRA	FRA	FRA	FRA	FRA	FRA
Shortstop	ND	ND	ND	ND	ND	ND
Antioxidant	NST	ST	ST	NST	ST	ST
Catalyst	OHP	OHP	OHP	OHP	OHP	OHP
Emulsifier	FA	RA	RA	RA	RA	Mixed
Bound Styrene, per cent	23.5	23.5	23.5	23.5	23.5	23.5
Conversion, per cent	60	60	60	60	60	60
Mooney Viscosity	32	56[c]	62[c]	50[c]	68[c]	48[c]
Coagulation	SA	A	SA	SA	SA	AC
Carbon Black:						
Type	...	HAF	HAF	FEF	ISAF	HAF
PHR	...	52	60	60	75	75
Oil:						
Type	...	HI-AR	HI-AR	Naph	AR	HI-AR
PHR	...	10	10	37.5	37.5	50
Finishing	Normal	Normal	Normal	Normal	Normal	Normal

ing tentatives under its jurisdiction. Many of them are under consideration for revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

The following are the highlights of committee activity. No attempt has been made to report all the details of all subcommittees.

Subcommittee VI on Packings (R. F. Anderson, chairman) continues its work on additional test methods for "O" rings to be included in Method D 1147. Test methods for properties at elevated temperatures are being considered.

Subcommittee VIII on Nomenclature (R. G. Seaman, chairman) has made progress on the subject of definitions of Rubber, Rubber-Like, and Rubber Products. The proposed definitions[9] need to include quantitative values on extension and retraction. Work is under way in several laboratories to obtain these data.

Subcommittee X on Physical Testing (L. V. Cooper, chairman) has been enlarged to include jurisdiction over hardness, set, creep, tear and abrasion. Hardness, Set and Creep (formerly under Subcommittee XVII) is now Section I, with William H. King as chairman. Section II, with W. A. Rodgers as chairman, will handle Methods for Stress-Strain Determinations at Elevated Temperatures. Section III, with Frank Roth as chairman, will prepare Methods for Use of Strain Gage Machines. Section IV, with W. S. Scoville, Jr., as chairman, will consider the evaluation of Tear Test Methods.

Subcommittee XI on Chemical Analysis (W. P. Tyler, chairman) continues to review all analytical procedures proposed by Committee D-11 subcommittees and ISO/TC 45. It has completed work on the combination of Methods D 297, Chemical Analysis of Rubber Products, and Methods D 833, Identification and Quantitative Analysis of Synthetic Elastomers, into one tentative.

Subcommittee XII on Crude Natural Rubber (L. G. Mason, chairman) has under way an extensive program to determine the application of the test methods described in Methods D 1278 for the examination of various grades of natural rubber.

Subcommittee XIII on Synthetic Elastomers (B. S. Garvey, Jr., chairman) is continuing its work on additional test methods for synthetic rubbers. Section I on Sampling of Carbon Black Masterbatches, and Section II on Chemical Tests are working on methods for determination of oil in oil masterbatches. Section III on Physical Tests is working on the revision of the recipes for various synthetic rubbers. Work is also under way on the classification and description of extender oils.

Subcommittee XV on Life Tests (G. C. Maassen, chairman) has completed a 12-yr study of shelf aging of rubber compounds in comparison with accelerated aging. These data are now being analyzed statistically for the preparation of a comprehensive report on the correlation of oven and shelf aging.

Subcommittee XVI on Classification and Specification of Rubber Compounds (J. F. Kerscher, chairman) continues to work jointly with Section IV-D of the SAE-ASTM Technical Committee on the development of the "Expanded Tabular System" for the classification of rubber compounds. This new system will augment or replace Specifications D 735, making it applicable to all segments of the rubber industry for both national and international purposes.

---

[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[9] See Editorial Note, p. 360.

Subcommittee XX on Adhesion Tests (H. H. Irvin, chairman) is now considering a study of environmental effects such as temperature and conditioning media on the adhesion of rubber to rigid materials. It is also considering a method for measuring adhesion under dynamic conditions.

Subcommittee XXI on Testing of Rubber Cements and Related Products (J. F. Anderson, chairman) has two sections studying methods of measuring the adhesion of textile and wire cord for tire use.

Subcommittee XXIV on Coated Fabrics (K. L. Keene, chairman) now has under consideration methods for cold-crack resistance and flame resistance of coated fabrics.

Subcommittee XXV on Low-Temperature Tests (R. S. Havenhill, chairman) is considering a gravity method as an alternate to the solenoid method for measuring low-temperature brittleness. It is cooperating with Committee D-20 on Plastics on this method.

Subcommittee XXVI on Processing (R. H. Taylor, chairman) is conducting a study of a standard extrusion die for evaluating the extrusion properties of rubber compounds, hoping to develop a standard procedure.

Subcommittee XXIX on Compounding Ingredients (A. E. Juve, chairman) is now working on a complete revision of Methods D 15, Sample Preparation for Physical Testing. A new section has also been established to develop methods for measuring the degree of dispersion of blacks and pigments in rubber.

This report has been submitted to letter ballot of the committee which consists of 265 members; 162 members returned their ballots, of whom 102 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. COLLIER,
Chairman.

JOHN J. ALLEN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Definitions of Terms Relating to:

Rubber and Rubber-like Materials (D 1566 – 58 T).

Revision of Tentative Methods for:

Chemical Analysis of Synthetic Elastomers (Solid Styrene-Butadiene Copolymers) (D 1416 – 58a T).

Revision of Tentative Recommended Practice for:

Description of Types of Styrene-Butadiene Rubbers (SBR) (D 1419 – 60 T), and
Description of Types of Styrene-Butadiene Rubber (SBR) and Butadiene Rubber (BR) Latices (D 1420 – 59 T).

Revision of Tentative Specification and Methods of Test for:

Flexible Foams Made from Polymers or Copolymers of Vinyl Chloride (D 1565 – 59 T).

The revision of Tentative Definitions D 1566 was accepted by the Standards Committee on September 29, 1960, and the other recommendations were accepted on December 20, 1960. They appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year: on March 14 and 15, 1960, in New York, N. Y. Six papers and an analytical demonstration were presented at the meetings: "Compatibility of Silicate and Phosphate Builders for Liquid Detergents" by R. Getty and W. Stericker; "Demonstration of Phosphate Analysis by the Auto Analyzer Technique" by D. P. Lundgren; "The Estimation of Soil Redeposition" by A. R. Martin and R. C. Davis; "Micelle Formation and Water Exchange Between Fabric and Micelles in Drycleaning Systems" by E. Hirschhorn; "Testing of Drycleaning Detergents of the Charge Type" by J. B. Schapiro; and "Laboratory Methods for Evaluation of Drycleaning Detergents" by L. E. Weeks and J. T. Lewis.

The Committee D-12 Award for outstanding performance in the field of soaps and detergents was presented to A. M. Schwartz by Award Committee Chairman E. W. Blank in recognition and appreciation of outstanding contributions and service in the field of detergents and detergency. Dr. Schwartz has been chairman of several of the technical subcommittees of Committee D-12 and is currently chairman of Subcommittee G-2 on Nomenclature and Definitions.

The committee consists of 117 members, of whom 58 are classified as producers, 32 as consumers, and 27 as general interest members, with 6 consulting members.

The officers elected for the ensuing term of two years are as follows:

Honorary Chairman, F. W. Smither.
Chairman, J. C. Harris.
Vice-Chairman, E. W. Blank.
Secretary, H. R. Suter.
Members of Advisory Committee G-1: R. E. Hauber, W. H. Joy, D. Price, A. M. Schwartz, W. Stericker, and A. R. Martin.

Accepted with regret, and with full appreciation of their accomplishments, were the resignations of J. R. Wiebush and M. G. Kramer, former chairman of Task Groups T-3 on Drycleaning and T-5 on Physical Methods, respectively. Newly appointed chairmen for these subcommittees are A. R. Martin and J. L. Darragh, respectively.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the proposed Tentative Method for Measuring Redeposition of Soil on Cotton Fabric.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[2]

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] This proposed method appears in the 1960 Compilation of ASTM Standards on Soaps and Other Detergents.

[2] The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 10.

Tentative Method for:

Determination of Ethylene Diamine Tetraacetate (EDTA) in Soap or Synthetic Detergents, and

Test for Sodium Alkylbenzene Sulfonate in Synthetic Detergents by Ultraviolet Absorption.

REVISIONS OF TENTATIVES

The committee recommends revisions as appended hereto[2] of the following tentatives and their continuation as tentative:

Tentative Methods for:

Sampling and Chemical Analysis of Alkylbenzene Sulfonates (**D 1568 – 58 T**), and

Sampling and Chemical Analysis of Fatty Alkyl Sulfates (**D 1570 – 58 T**).

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[3] of the Standard Specifications for Liquid Toilet Soap (**D 799–51**)[4] and reversion of the specifications to tentative.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Trisodium Phosphate (**D 538 – 57 T**).[4]

Tentative Method of Test for:

Copper in Soaps and Soap Products (**D 460 – 57 T**).[4]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Definitions of:

Terms Relating to Soaps and Other Detergents (**D 459 – 58 T**).

Tentative Method of:

Testing Detergent Cleaners for Evaluation of Corrosive Effects on Certain Porcelain Enamels (**D 1567 – 58 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Specifications for Borax (D 929 – 50):[4]

Section 3.—In the table of requirements, change the words "Free acid" to "Excess acidity," and the words "Free alkali calculated as NaOH, max, per cent . . . 0.25" to "Excess alkalinity as Na_2O, max, per cent . . . 0.2."

Standard Methods of Sampling and Chemical Analysis of Alkaline Detergents (D 501 – 58):[4]

Section 84.—Change the center heading preceding this Section from "Total Borate and Total Alkalinity or Acidity" to read "Total Borate and Excess Alkalinity or Acidity."

Section 86.—In the second and third lines, change ". . . and of free alkali or free acid . . . " to read ". . . and of excess alkalinity calculated as Na_2O or excess acidity . . . ." Also change calculation (*1*) to read: "(*1*) If AN_A is greater than BN_B, excess alkalinity, calculated as Na_2O, per cent = $(AN_A - BN_B) \times 1.55$." Also under (*2*), change "Free acid" to "Excess acidity."

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

---

[3] The revised specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 10.

[4] 1958 Book of ASTM Standards, Part 10.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee S-2 on Specifications for Soaps and Synthetic Detergents (W. H. Joy, chairman):

Task Group 1 on Synthetic Detergents (R. L. Liss, chairman) will prepare a specification on alkylaryl sulfonates.

Task Group 2 on Scouring Powders (C. A. Gerardi, chairman) was organized to work on specifications for scouring powders.

Subcommittee T-2 on Analysis of Soaps and Synthetic Detergents (J. C. Harris, chairman):

Task Group 1 on Classification of Surfactants (H. C. Speel, chairman) proposed that the system of classification developed by Schwartz, Perry, and Berch (Vol. 2 of Surface Active Agents and Detergents) be followed, with modification if indicated.

Task Group 3 on Bibliographical Abstracts of Analytical Methods for Surfactants (J. C. Harris, chairman).—A new supplement to the Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (*STP No. 150*), including over 200 new references, is in preparation.

Task Group 4 on Separation of Active Ingredient (R. M. Kelley, chairman) will evaluate methods published in the literature, preliminary to development of ASTM methods.

Task Group 5 on Infrared Determination of Surfactants (E. A. Setzkorn, chairman) is working in conjunction with Task Group 16, using the same samples for evaluation.

Task Group 8 on Determination of CMC (J. L. Darragh, chairman) is continuing work on a method.

Task Group 16 on Qualitative Method for Identification of Surfactants and Organic Builders (L. E. Weeks, chairman).—Further cooperative tests for refinement of procedure are planned for the coming year.

Task Group 20 on Total Active Ingredient (C. A. Cohen, chairman).—A procedure for separation by extraction should be completed within the coming year.

Task Group 21 on Detergent Alkylate (J. C. Harris, chairman):

Subgroup A on Distillation Range (J. L. Darragh, chairman).—Following reevaluation of Committee D-2 procedures, it is proposed that Methods D 447 or Method D 86 be adopted for use in distillation procedures.

Subgroup C on Acid Wash Color (R. C. Stillman, chairman).—A procedure has been adapted from other ASTM work, but use of the method will be deferred pending discussion of apparatus and procedure.

Subgroup D on Standard Sulfonation Procedure and Analysis cf Product (C. A. Cohen, chairman).—As support for these procedures, over 30 pure alkylbenzene sulfonates have been synthesized and analyzed. Methods are in preparation.

Task Group 22 on Estimation of Ethylenediamine Sodium Tetraacetate (E. W. Blank, chairman) recommended for publication as tentative the Method for Determination of Ethylene Diamine Tetraacetate in Soap or Synthetic Detergents referred to earlier in the report. Procedures adapted to other sequestrants will be investigated during the coming year.

Task Group 23 on Phosphate Analysis of Surfactant Compositions by Paper Chromatography (C. F. Callis, chairman) is developing improved procedures.

Task Group 24 on Determination of Chemical Bleaching Agents (C. H. Schmiege, chairman). — Tentative methods for analysis of sodium and calcium hypochlorite and of chlorocyanuric acids and derivatives are being prepared. Following the chlorine-containing bleaches, those containing active oxygen will be investigated.

Subcommittee T-5 on Physical Testing (M. G. Kramer, chairman):

Task Group 1 on Measurement of Soil Redeposition (A. R. Martin, chairman) developed the Method for Measuring Redeposition of Soil on Cotton Fabric for publication as information only as noted earlier in the report. Development of a method using clay instead of carbonaceous soil will be undertaken.

Task Group 2 on Measurement of Wetting Power of Surfactants (C. A. Gerardi, chairman).—An investigation of the Draves skein and Shapiro tape methods is being made; results are still inconclusive as to which is the most precise.

Task Group 3 on Measurement of Reflectance of Test Fabrics (R. L. Liss, chairman).—A test method based on previous work will be submitted to task group members along with test cloth and white and gray standards for cooperative testing.

Task Group 5 on Brightener Evaluation (M. S. Furry, chairman).—A survey of T-5 members was made to define the scope of this program. It was decided to first study buildup, then substantivity and light and bleach stability. Members will be solicited for methods covering these items.

Task Group 9 on Methods for Determination of the Cloud Point of Detergents (F. E. Woodward, chairman) is evaluating methods in current use.

Task Group 10 on Methods for Evaluation of Detergents in Dishwashing (R. E. Johnson, chairman) is attempting to develop tests to evaluate any detergent in a given dishwasher or any dishwasher with a given detergent.

Task Group 11 on Studies of Primary Deposition of Soil (A. M. Schwartz, chairman).—A bibliography on this subject has been completed and will be submitted for acceptance as a Special Technical Publication.

This report has been submitted to letter ballot of the committee, which consists of 117 members; 87 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. C. HARRIS,
Chairman.

H. R. SUTER,
Secretary.

REPORT OF COMMITTEE D-13
ON
TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two meetings during the year: in New York City, Oct. 13–16, 1959, and on March 1–4, 1960. In addition, several special committees, subcommittees, and task groups held sessions at intervals throughout the year.

At the March meeting the Eleventh Award of the Harold DeWitt Smith Memorial Medal was presented to Werner von Bergen, Associate Director of Research, J. P. Stevens and Co., in recognition of his many contributions to fiber science.

In addition to the regular achievements of establishing new standards and revisions of existing ones, Committee D-13 completed the task of reviewing and bringing up to date the regulations governing the work of the committee.

Subcommittee B-6, under the leadership of Arthur Scroggie, successfully arranged for a delegation of 15 representatives to participate in the plenary session of ISO TC/38 on Textiles to be held in London, England, May 19 to 28, 1960. Committee D-13 assumes a major part of the responsibility of the U. S. textile industry in establishing International Standards for Textile Materials this year, as it has done in earlier sessions in 1948, 1952, and 1956. Kenneth Hertel will head the United States Delegation.

Committee D-13, in conjunction with ASA Sectional Committee L-23, has prepared a proposal on "Introduction of the Tex System for Designation of Yarn Number" for consideration by the textile industry in the United States. An outline of the plan, a reprint from the ASTM BULLETIN, is being distributed to some 4000 people in the industry.

A new subcommittee section to deal with standardization problems in automotive fabrics was created during the year. Membership will include representatives from the producer, consumer, and general interest groups. Mr. Jackson Bauer was appointed chairman. Mr. J. B. Curley was appointed chairman of Subcommittee B-8 to replace Mr. D. S. Hamby.

Mr. William D. Appel was elected an Honorary Member of Committee D-13 in recognition of his outstanding leadership in the work of the committee and for his accomplishments in establishing and maintaining close liaison with other organizations.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. F. Schiefer.
First Vice-Chairman, D. S. Hamby.
Second Vice-Chairman, B. S. Sprague.
Third Vice-Chairman, M. C. Bullock.
Secretary, H. A. Ehrman.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 1 new standard, 9 new tentatives, the revision of 6 tentatives, the tentative revision of 2 standards, the immediate adoption of revisions in 3 standards, the revision and reversion to tentative of 3 standards, and the withdrawal of 2 tentatives.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

The standards and tentatives affected, together with the revisions recommended, are listed in detail in the Appendix.[1]

All other standards and tentatives not specifically referred to are being studied by the respective subcommittees and it is recommended that they be continued in their present status.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 386 voting members; 158 members returned their ballots, of whom 126 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. L. WHITTIER,
Chairman.

H. A. EHRMAN,
Secretary.

---

[1] See p. 367.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials. These standards and tentatives appear in their present form in the 1958 Book of ASTM Standards, Part 10, and its 1959 Supplement.

NEW STANDARD

The committee recommends for publication as a separate standard the Method of Test for Linear Density of Cotton Fibers (Array Sample) as appended hereto,[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the method may be referred to letter ballot of the Society. The method in its previous form constituted a part of Standard Method D 1442 – 54.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of Test for:

Neps in Wool Top,
Sample Reweighing of Lots of Packaged Raw Wool,
Conditioning Textiles and Textile Products for Testing,
Imperfection Count of Textile Yarns,
Elastic Fabrics, to replace Methods D 1333 – 54 T and D 1377 – 55 T,
Thickness of Textile Materials, to replace eventually Section 5 of Methods D 39 – 59,
Dimensional Change of Knitted Fabrics (Adoption as tentative of Appendix X, Proposed Method of Test for Dimensional Change of Knitted Fabrics in Laundering and Dimensional Restorability of the Laundered Fabric),[2]
Elastic Properties of Uncrimped Fibers, and
Colorfastness to Commercial Laundering and to Domestic Washing of Tufted Rugs and Carpets.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods for Identification of Fibers in Textiles (D 276 – 59 T):[3]

Section 1.—Add in the proper alphabetical order the names "Creslan," "Darvan," "Kodel," and "Zefran." Also add the registered trademark identification as indicated in Table I.

Section 8 (d).—Add the registered trademark identification as indicated in Table I.

Table V.—Delete the "Refractive Index" column as these values are replaced by those in the new Table VII (the accompanying Table I).

New Table.—Add as new Table VII the accompanying Table I.

Figure 23.—Replace this figure with the accompanying Fig. 1.[4]

---

[1] The new standard and tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 10.

[2] 1959 Compilation of ASTM Standards on Textile Materials, p. 809.

[3] 1959 Supplement to Book of ASTM Standards, Part 10.

[4] The new figures are not included in this report but appear in the revised Methods D 276; see 1960 Supplement to Book of ASTM Standards, Part 10.

New Figures.—Add as new Figs. 32 to 35 the accompanying Figs. 2 to 5.[4]

Tentative Method of Test for Vegetable Matter and Alkali-Insoluble Impurities in Scoured Wool (D 1113 – 54 T):[5] Revise as appended hereto.[6]

Tentative Method of Test for Unevenness of Textile Strands (D 1425 – 59 T):[3] Revise as appended hereto.[6]

Tentative Method of Test for Recovery of Textile Fabrics from Creasing, Using the Vertical Strip Apparatus (D 1295 – 53 T):[5] Revise as appended hereto.[6]

Tentative Method of Test for Tuft Bind Pile Floor Coverings (D 1335 – 54 T):[5] *Section 1.*—Delete the last sentence.

Tentative Method of Test for Linear Density of Testile Fibers by the Vibroscope (D 1577 – 58 T):[5] Revise as appended hereto.[6]

TENTATIVE REVISION OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

Standard General Methods of Testing Woven Fabrics (D 39 – 59):[3]

Sections 3, 4, 6, 7, 8, and 18.—Replace Sections 3, 4, 6, 7, 8, and 18 with a reference to the Tentative Method of Test for Construction Characteristics of Woven Fabrics (ASTM Designation: D 39).

Section 5.—Replace Section 5 with a reference to the Tentative Method for Measuring Thickness of Textile Materials (ASTM Designation: D 1777).

Section 17.—Replace Section 17 with a reference to the Tentative Method of Test for Twist in Single Spun Yarns (Untwist-Twist Method) (ASTM Designation: D 1422), and to the Tentative Method of Test for Twist in Yarns (Direct-Counting Method) (ASTM Designation: D 1423).

Standard Definitions of Terms Relating to Textile Materials (D 123 – 59).[3]

Add the following three new tentative definitions to read as follows:

Wash-and Use, *adj.*—A term applied to fabrics and to household textiles, which can be washed and placed in normal service uses repeatedly with little or no ironing. (See Wash-and-Wear.)

Wear-Wrinkle Resistant (Wear-Crease Resistant), *adj.*—A term applied to textile fabrics which satisfactorily maintain their appearance by recovery from sharp folds imposed during wear. (See Wash-and-Wear, Note 1, and Wrinkle Resistance.)

Wash-Wrinkle Resistant (Wash-Crease Resistant), *adj.*—A term applied to textile fabrics which satisfactorily maintain their appearance by recovery from sharp folds imposed during laundering. (See Wash-and-Wear, Note 1, and Wrinkle Resistance).

Appendix V.—Replace Fig. 1 with the accompanying Fig. 6.[7]

New Appendix.—Add as Appendix VI the following Proposed Generic Names and Definitions for Manufactured Fibers, which have been taken from Rule 7, Federal Trade Commission Rules and Regulations under Textile Fiber Products Identification Act effective March 3, 1960.[7]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Specifications and Methods of Test for Cotton Goods for Rubber and Pyroxylin Coating (ASTM Designation: D 334 – 40):[5]

Section 1.—Change to read as follows:

---

[5] 1958 Book of ASTM Standards, Part 10.

[6] The revised methods appear in the 1960 Supplement to Book of ASTM Standards, Part 10.

[7] The new figure and appendix are not included in this report but appear in the revised Definitions D 123; see 1960 Supplement to Book of ASTM Standards, Part 10.

TABLE I.—THE REFRACTIVE INDEXES OF TEXTILE FIBERS.

(New Table VII of Methods D 276)

NOTE.—These values are for very specific fibers which explains the highly accurate values given. In identification work, such precision is not practical; values within 0.02 or 0.03 of those given will usually suffice.

Fiber	Refractive Indices		Birefringence	Source
	ϵ	ω	$\epsilon - \omega$	
Acetate (secondary)	1.479	1.477	0.002	*a*
Acrilan®	1.524	1.520	0.004	*a, b*
Arnel® (tertiary acetate)	1.472	1.471	0.001	*c*
Chlorinated wool	1.549	...	...	*d*
Cotton	1.580	1.533	0.047	*a*
Creslan®	1.517	1.521	−0.004	*c*
Cuprammonium rayon	1.552	1.520	0.030	*a*
	1.548	1.527	0.021	
Dacron®	1.710	1.535	0.175	*d*
Darvan®	1.484	1.476	0.008	*e*
Dynel®	1.536	1.531	0.005	*b*
Flax	1.596	1.528	0.068	*a*
	1.594	1.532	0.062	*f*
Fortisan	1.547	1.523	0.024	*c*
Fortisan 36	1.551	1.520	0.031	*c*
Glass	1.547	1.547	0.000	*b*
Hemp	1.581 to 1.591	1.521 to 1.530	0.055 to 0.065	*g*
Kodel®	1.632	1.534	0.098	*h*
Jute	1.577	1.536	0.041	*g*
Nylon 6	1.568	1.515	0.053	*a*
Nylon 66	1.547	1.521	0.026	*c*
	1.582	1.519	0.073	*f*
Orlon®	1.500 to 1.510	1.500 to 1.510	0.000	*a, b*
Polyethylene	1.556	1.512	0.044	*a*
Ramie	1.596	1.528	0.068	*a, f*
Saran®	1.60 to 1.63	1.60 to 1.63	0.000	*a, c*
Silk	1.591	1.538	0.053	*f, d*
Terylene®	1.710	1.535	0.175	*d*
Velon®	1.60 to 1.63	1.60 to 1.63	0.000	*f*
Verel®	1.538	1.538	0.000	*h*
Vicara®	1.536	1.532	0.004	*a, d*
	1.534	1.534	0.000	*c*
Viscose	1.544	1.520	0.024	*a*
	1.547	1.521	0.026	*d*
Wool	1.556	1.547	0.009	*c, f*
	1.553	1.547	0.011	*a*
Zefran®	1.512	1.512	0.000	*i*

1. *Scope.*—These specifications and methods of test only apply to cotton goods to be coated with either rubber or pyroxylin. They do not apply to those which contain rubber thread.

Section 5.—Change the heading to read "Sizing and Nonfibrous Materials," and revise the text to read as follows:

5. The maximum percentage of sizing and nonfibrous materials shall be as follows:

	Sizing, per cent	Nonfibrous Materials, per cent
Sheetings	6.50	10.75
Drills	7.75	12.0
Sateens	7.75	12.0
Broken twills	7.75	12.0
Osnaburgs	7.75	12.0

Section 6.—Change to read as follows:

6. The maximum amount of copper, manganese, and grease (chloroform extract) shall be as follows:

Copper[a]	0.001 per cent
Manganese	0.0015 per cent
Grease	1.0 per cent

[a] This limit stated for copper is not applicable to cotton goods that are printed or colored with phthalocyanine dyes. The suitability for such goods may be determined by accelerated aging of a suitable rubber or pyroxylin coating applied hereto.

Section 15.—Change the heading to read "Grease and Sizing," and in Paragraph (*a*) change "the amount of sizing" to read "the amount of grease and sizing." Revise the beginning of Paragraph (*d*) to read as follows:

---

[a] "Identification of Textile Materials," 4th Edition, The Textile Institute, London.

[b] A. N. J. Heyn, "Observations of the Birefringence and Refractive Index of Synthetic Fibers with Special Reference to Their Identification," *Textile Research Journal*, Vol. 22, No. 8, p. 513 (1952).

[c] "Fibers Chart," *Modern Plastics*, Encyclopedia Issue (1958).

[d] H. R. Mauersberger, "Matthews' Textile Fibers," 6th Edition.

[e] B. F. Goodrich Chemical Co., Avon Lake, Ohio.

[f] Milton Harris, "Handbook of Textile Fibers," 1st Edition.

[g] Bruns Luniak, "The Identification of Textile Fibers," Sir Isaac Pitman and Sons, Ltd., London.

[h] Eastman Chemical Products, Inc., Kingsport, Tenn.

[i] Dow Chemical Co., Midland, Mich.

> (*d*) *Procedure.* — (*1*) *Grease.* — Extract the specimen described in Paragraph (*c*) with chloroform, U.S.P., for a minimum of 20 extractions in a Soxhlet extraction apparatus. Evaporate the solvent from the extract in a tared flask at a temperature of 79 to 81 C to constant weight. Cool the dried extract and flask in a desiccator, and then weigh. This weight less the tare of the flask gives the grease, or chloroform soluble material.
>
> (*2*) *Sizing.*—Determine the sizing in the extracted sample as follows:
>
> Prepare an approximately 0.1 *N* solution by diluting....

In Paragraph (*e*) change the reference to Paragraph (*d*) to read "Paragraph (*d*), Item (2)."

Standard Methods of Test for Small Amounts of Copper and Manganese in Textiles (D 377 – 54):[5] Revise as appended hereto.[6]

Standard Method of Test for Strength of Cotton Fibers (Flat Bundle Method) (D 1445 – 57):[5] Revise as appended hereto,[6] which revision also includes Appendix XII, Method of Test for Strength of Cotton Fibers Using a Finite Gage Length (Flat Bundle Method).

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Definitions of Terms Relating to Textile Materials (D 123 – 59):[3]

Part A.—Replace the definition of "Tape, woven, *n*" with the following definition:

> **Tape, Woven, Non-Elastic,** *n.*—A woven narrow fabric, made principally of natural, and/or man-made or monofilament yarns, but not containing rubber or other similar elastic strands, and weighing less than 15 oz per sq yd (2.604 lb per 100 yd per in. of width).

Part B. Appendix I.—Replace the definition of "Webbing, Non-Elastic, *n*" with the following definition:

> **Webbing, Woven, Non-Elastic,** *n.*—A woven narrow fabric, made principally of natural, and/or man-made or monofilament yarns, but not containing rubber or other similar elastic strands, and weighing more than 15 oz per square yard (2.604 lb per 100 yd per in. of width).

Standard Specifications and Methods of Test for Asbestos Yarns (D 299 – 54):[5]

Section 11(*b*) *and* (*c*).—Change the factor "0.90" in both formulas to "0.95." Revise the note to read as follows: "NOTE.—The factor 0.95 in the formula compensates for an average contraction of 5 per cent incurred in twisting."

Standard Method of Test for Fiber Weight per Unit Length and Maturity of Cotton Fibers (Array Method) (D 1442 – 54).[5] Revise as appended hereto.[6] The test for linear density of cotton fibers (array sample) which previously constituted a part of this method is recommended for publication as a separate standard as indicated above.

WITHDRAWAL OF TENTATIVES

The committee recommends the withdrawal of the following tentatives:

Tentative Methods of Testing:

Elastic Fabrics (**D 1333 – 54 T**), and

Wide Elastic Fabrics (**D 1377 – 55 T**), as these methods are replaced by the proposed Tentative Method of Testing Elastic Fabrics.[1]

REPORT OF COMMITTEE D-14
ON
ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year: at Case Institute of Technology, Cleveland, Ohio, on November 5–6, 1959, and at the Sheraton Hotel, Philadelphia, Pa. on March 31 and April 1, 1960.

At the fall meeting, Mr. J. A. Hrones, Vice-President for Academic Affairs of the Case Institute of Technology, spoke on "Changing Emphases in Engineering Education." At the spring meeting, Mr. R. P. Havilland of the Missile, Satellite and Space Vehicle Dept. of the General Electric Co. spoke on "Physical Basis of Failures."

The committee consists of 138 members, of whom 51 are classified as producers, 62 as consumers, and 25 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. E. Rutzler, Jr.

Vice-Chairman, A. A. Marra.

Secretary, G. S. Casebolt.

Membership Secretary, R. M. Kell.

Research Reports File.—On March 28, 1960, Committee D-14 on Adhesives dedeposited the following report in the Society's Research Reports File at Headquarters: File No. RR 4:D-14, "Effect of Simulated Service Tests on Adhesive Bonds" by Frank W. Reinhart. (Approved and submitted by John E. Rutzler, Jr., chairman.)

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Determining the Water Absorptiveness of Fiberboard Specimens for Adhesives (D 1714 – 60 T), and

Bonding Permanency for Water or Solvent Soluble Liquid Adhesives for Automatic Machine Sealing Top Flaps of Fiberboard Specimens (D 1713 – 60 T).

These recommendations were accepted by the Standards Committee on April 14, 1960. The new methods of test will appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

NEW TENTATIVES

The committee recommends for publication as tentative the following method, specification and recommended practice as appended hereto:[1]

Tentative Method for:

Climbing Drum Peel Test for Adhesives.

Tentative Specification for:

Adhesive for Acoustical Materials.

Tentative Recommended Practice for:

Conducting Creep Tests of Metal-to-Metal Adhesives.

*** Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.**

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Consistency of Adhesives (**D 1084 – 55 T**),[2]
Adhesives Relative to Their Use as Electrical Insulation (**D 1304 – 54 T**),[2]
Amylaceous Matter in Adhesives (**D 1488 – 57 T**),[2]
Nonvolatile Content of Aqueous Adhesives (**D 1489 – 57 T**),[2]
Filler Content of Phenol, Resorcinol, and Melamine Adhesives (**D 1579 – 58 T**),[2]
Bonding Permanency of Water or Solvent Soluble Liquid Adhesives for Labeling Glass Bottles (**D 1581 – 58 T**),[2]
Nonvolatile Content of Phenol, Resorcinol and Melamine Adhesives (**D 1582 – 58 T**),[2] and
Water Absorptiveness of Paper Labels (**D 1584 – 58 T**).[2]

Tentative Specifications for:

Adhesives for Automatic Machine Labeling of Glass Bottles (**D 1580 – 58 T**).[2]

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that all tentative revisions of Standard Definitions of Terms Relating to Adhesives (**D 907 – 55**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Strength Properties of Adhesives in Shear by Tension Loading (Metal-to-Metal) (**D 1002 – 53 T**),
Effect of Moisture and Temperature on Adhesive Bonds (**D 1151 – 58 T**),
Susceptibility of Dry Adhesive Films to Attack by Roaches (**D 1382 – 55 T**),
Susceptibility of Dry Adhesive Films to Attack by Laboratory Rats (**D 1383 – 55 T**),
Nonvolatile Content of Urea-Formaldehyde Resin Solutions (**D 1490 – 57 T**), and
Measuring the Hydrogen Ion Concentration of Dry Adhesive Films (**D 1583 – 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Tests (Carl Dahlquist, chairman) has completed work on the T-peel test and is now submitting it to subcommittee letter ballot.

The subcommittee completed the proposed Method for Climbing Drum Peel Test for Adhesives and the proposed Recommended Practice for Conducting Creep Tests for Metal-to-Metal Adhesives referred to earlier in this report. The subcommittee is balloting on a method for impact testing of adhesives in torsional shear, cleavage, and tension.

Subcommittee II on Analytical Tests (G. F. Lipsey, chairman) has recommended the advancement to standard of Methods D 1488 and D 1489. A Method of Test for Density of Adhesives in Fluid Form has been approved by committee letter ballot for submission to the Standards Committee as a tentative method. The subcommittee is now working on a method of test for the determination of the flash point of adhesives.

Subcommittee III on Tests for Permanency (H. R. Butzlaff, chairman) is revising Method D 1183 to include adherends other than wood, following the results of an interlaboratory study.

---

[2] 1958 Book of ASTM Standards, Part 6.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Recommended practices for (1) determining the resistance of plywood adhesives to mold and (2) resistance of adhesive bonded joints to high-energy radiation are being balloted by the committee. Proposed practices for (1) atmospheric exposure of adhesive bonded structures and (2) exposure of adhesive bonded specimens under stressed conditions are being prepared.

Subcommittee IV on Working Qualities (N. J. DeLollis, chairman) has in preparation methods on pressure-sensitive tack, for flow properties of adhesives, and for the determination of penetration.

Subcommittee V on Specifications (M. Petronio, chairman) is preparing specifications for polyvinyl acetate resin emulsion adhesives and for general purpose (mending) adhesives, and has completed the proposed Tentative Specification for Adhesives for Acoustical Materials referred to earlier in the report.

Specifications on top flaps, water absorptiveness, and bonding permanency of adhesives for fiberboard shipping cases are in preparation.

Tentative Methods D 1580, D 1581, and D 1584 are being recommended for advancement to standard.

Subcommittee VI on Nomenclature and Definitions (F. H. Wetzel, chairman) prepared definitions of "throwing," "cobwebbing," "cottoning," "feathering," "mastic," and "body" for advancement to standard; definitions of "adhesive failure," "cohesive failure," "adherend failure," "contact failure," "supported adhesive film," and "unsupported adhesive film" for committee consideration; and definitions of "maturing temperature," "foamed adhesive," "fillet," "telegraphing," and "photographing" for subcommittee consideration.

Subcommittee VII on Research Problems (C. B. Hemming, chairman) has held discussions as to how this subcommittee might best serve Committee D-14. The subcommittee expects to hold at least one talk or panel-audience discussion at each meeting. It was also proposed that a Manual on the Testing of Adhesives be written to inform people of (1) intent of precautions in test methods, (2) the general utility of various tests, and (3) the pitfalls to be encountered by the uninitiated.

Subcommittee VIII on Electrical Properties of Adhesives (F. Moser, chairman) has discussed the need for methods of measuring the electrical resistance of conductive adhesives and has set up a new section to cover this work. Also, methods of test for the corrosive properties of adhesives under electrical stress are to be reviewed, and invitations are to be sent out requesting membership on the new section.

Subcommittee X on Wood Adhesives (R. F. Blomquist, chairman) is now comprised of two groups meeting and working independently, one on the West Coast and one in the East and Middle West, but coordinated through the main subcommittee. Each group has been actively reviewing existing shear and tensile test procedures for wood adhesives, and some revisions are under way. Extensive reviews are being made on the problems involved in better evaluation of adhesives for lumber, veneer, assembly of wood components, and for composites of wood with other materials as a basis for more intelligent consideration of new and improved test procedures. Special attention has been given to evaluation of amounts and type of wood failure in broken bonds, and in the evaluation of binders for particle board.

Subcommittee XI on Metal Bonding Adhesives (R. L. Patrick, chairman) is studying surface preparation, fatigue, durability, and in-process inspection of bonded metal structures and specimens preparatory to writing test methods. It has worked with Subcommittee I on impact tests by tensile, cleavage, and torsional shear methods. Proposed methods for static cleavage and surface cleanliness are being prepared for committee action.

The subcommittee is now functioning as two interdependent groups, with the West Coast Task Group under the direction of Steven Yurenka. Definition of the areas of effort of Subcommittee XI is being worked out with Subcommittee I. There will be a combined effort in certain areas: disk shear, impact cleavage, shear tests, and so on.

The West Coast Task Group is reviewing existing test methods and developing new methods for lap and disk shear, compression and tension creep, high-temperature testing, metal surface preparation, and inspection and process control.

Membership of the West Coast group is drawn largely from the research staffs of West Coast aircraft manufacturing companies and local adhesives companies.

A Symposium on Adhesion and Adhesives was held during the Third Pacific Area National Meeting at San Francisco, Calif.; the papers presented will be published as *ASTM STP No. 271.*

Subcommittee XII on Adhesives for Plastics (D. A. George, chairman) will send a recommended practice for surface preparation of plastic adherends prior to bonding to letter ballot of the subcommittee before the fall meeting.

The existing test methods are being reviewed for possible modification so they can be applied to adhesive bonds made with plastic adherends. The present method of test for tensile shear strength of adhesive bonds with metal adherends is now being revised for use with plastic adherends.

The need for new methods of test specifically for use with plastic adherends is now being investigated.

Subcommittee XIII on Flooring Adhesives (C. W. Bayley, Jr., chairman) is preparing methods of tests for (1) strength in tensile shear of "above grade" flooring adhesives, (2) resistance of "above grade" flooring adhesives to soap and detergents, and (3) temperature resistance of "above grade" flooring adhesives. A general procedure for preparing standard concrete slabs for use as test substrates has been discussed. The subcommittee chairman has been authorized to prepare for subcommittee ballot methods of test for test of "on grade" flooring adhesives using standard concrete substrates.

This report has been submitted to letter ballot of the committee, which consists of 138 members; 66 members returned their ballots, of whom 64 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. E. RUTZLER, JR.,
Chairman.

J. J. LAMB,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards tentative revisions of Standard Definitions of Terms Relating to Adhesives (T 907 – 55). The tentative revisions were accepted by the Standards Committee on September 29, 1960, and appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

REPORT OF COMMITTEE D-15
ON
ENGINE ANTIFREEZES[*]

Committee D-15 on Engine Antifreezes held one meeting during the year: on April 5, 1960 in Washington, D. C. Subcommittee meetings were held on November 4 and 5, 1959, in New York City and on April 4 and 5, 1960, in Washington, D. C.

Three members were elected to membership in Committee D-15 during the year. The committee now has 51 participating members. There are 40 voting members, of whom 13 are classified as producers, 17 as consumers, and 10 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. E. Vogel.
Vice-Chairman, F. G. Church.
Secretary, W. D. McMaster.
Advisory Committee Members-at-Large: V. O. Hatch and R. W. Kallgren.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Hydrometer-Thermometer Field Tester for Engine Antifreezes (**D 1124 – 58 T**).

Tentative Method for:

Glassware Corrosion Test for Engine Antifreezes (**D 1384 – 55 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Antifreeze Field Testers (F. G. Church, chairman) reviewed the proposed revision of the Federal Specification GG-H-935, "Testers, Antifreeze Solutions."

Subcommittee III on Physical Properties (C. M. White, chairman) is preparing a method of test for determining the effect of antifreeze solutions on organic finishes for automotive vehicles.

Subcommittee IV on Chemical Properties (E. F. Harford, chairman) is investigating a simple method of glycol analysis based on gas chromatography.

Subcommittee V on Effect of Antifreeze on Rubber Hose (O. W. Chandler, chairman) is studying a hose burst test method for evaluating the effect of antifreezes on cooling system hoses.

Subcommittee VI on Simulated and Actual Service Testing (C. H. Sweatt, chairman).—The Study Group on Glassware Test Methods (E. L. Gibson, chairman) is continuing the development of a glassware method of test for foaming of engine antifreezes. It is also completing collaborative tests to evaluate proposed changes in the glassware corrosion test (ASTM Method D 1384 – 55 T).

The Study Group on Service Simulation Test Methods (V. O. Hatch, acting chairman) is conducting collaborative tests on a bench-type circulating test to evaluate corrosion and foaming properties under simulated service conditions. The apparatus consists of the basic components of an automotive cooling system, including a cast iron reservoir, an automotive radiator and coolant pump.

---

[*] Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

Subcommittee VII on Specifications (B. E. Tiffany, chairman) is working on a recommended practice for purchasing engine antifreezes.

This report has been submitted to letter ballot of the committee, which consists of 40 voting members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. VOGEL,
Chairman.

C. O. DURBIN,
Secretary.

REPORT OF COMMITTEE D-16

ON

INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS*

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials and its subcommittees held two meetings during the year: on June 24 to 26, 1959, in Atlantic City, N. J., and on Feb. 3 to 5, 1960, in Chicago, Ill.

Two committee members passed away since the last report: L. J. Waldbauer and J. P. Graham.

The committee consists of 73 members, of whom 64 are voting members; of these, 32 are classified as producers, 23 as consumers, and 9 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. E. Sisco.
Vice-Chairman, M. Mitchell.
Secretary, K. H. Ferber.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the proposed Recommendations for Interlaboratory Testing of Industrial Aromatic Hydrocarbons and Related Materials.[1]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration (**D 1491 – 57 T**),[2] and

Bromine Index of Aromatic Hydrocarbons by Coulometric Titration (**D 1492 – 57 T**).[2]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Solidification Point of Benzene (**D 852 – 47**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

New Sections.—Add new Sections 2 and 3 to read as follows, renumbering subsequent sections accordingly:

2. *Outline of Method.*—Solidification point is measured by noting the maximum temperature reached during a controlled cooling cycle after the appearance of a solid phase.

3. *Definition.*—*Solidification Point* is an empirical constant defined as the temperature at which the liquid phase of a substance is in approximate equilibrium with a relatively small portion of the solid phase.

NOTE 1.—Solidification point is distinguished from freezing point which is described in the Method of Test for Measurement of Freezing Points of High-Purity Compounds for Evaluation of Purity (ASTM Designation: D 1015).

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] See Appendix, p. 381.

[2] 1958 Book of ASTM Standards, Part 8.

An interpretation of mol per cent purity in terms of freezing point is given in the Method of Test for Determination of Purity from Freezing Points of High-Purity Compounds (ASTM Designation: D 1016).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Industrial 90 Benzene (**D 837 - 56 T**).[2]

Tentative Method for:

Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (**D 1555 - 58 T**).[3]

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives and standards:

Standard Method of Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 - 56):[2]

Title.—Change the title to read: "Standard Method of Test for Distillation of Industrial Aromatic Hydrocarbons and Related Materials."

Section 1.—Revise the scope to read as follows:

> 1. *Scope.*—This method of test is intended for use in the distillation of industrial aromatic hydrocarbons and related materials of relatively narrow boiling ranges between 30 and 250 C.

Standard Methods of Test for Specific Gravity of Industrial Aromatic Hydrocarbons (D 891 - 59):[4]

Title.—Change to read: "Standard Methods of Test for Specific Gravity of Industrial Aromatic Hydrocarbons and Related Materials."

Section 1 (a).—Change the first sentence to read: "These methods are intended for use in determining the specific gravity (Note 1) of industrial aromatic hydrocarbons and related materials."

Section 7 (b).—Replace the last sentence with the following: "The height of the cylinder shall be such that after equilibrium has been reached the lowest point on the hydrometer will be at least 2.5 cm (1 in.) off the bottom of the cylinder."

Section 8.—Add the words, "and discard the rinse liquid" at the end of the second sentence.

Tentative Method of Test for Solidification Point of Aromatic Hydrocarbons and Related Materials (D 1493 - 58 T):[2]

Section 6.—In Paragraph (*d*) change the second sentence to read: "The maximum temperature reading is the observed solidification point (see Section 7)."

Add new Paragraphs (*e*) and (*f*) to read as follows:

> (*e*) Correct the observed solidification point for scale error of the thermometer and for the temperature of the emergent column, provided it differs from the stem temperature at which the thermometer was calibrated. Use the following equation:
>
> $$\text{Emergent column correction} = Kn(t_s - t_o)$$
>
> where:
>
> K = differential expansion coefficient of the liquid column and the glass of which the thermometer is made. For Centigrade mercury-in-glass thermometers, the value of K is 0.00016.
>
> n = number of degrees Centigrade emergent from the sample,
>
> t_s = specified average temperature of the liquid column of n degrees, and
>
> t_o = observed average temperature of the liquid column of n degrees.
>
> t_s and t_o should be determined by identical procedures during calibration and during use in this method.
>
> (*f*) Where specified, correct for the effect of known moisture content.

Section 7.—Change the first sentence

---

[3] 1958 Book of ASTM Standards, Parts 7 and 8.

[4] 1959 Supplement to Book of ASTM Standards; Part 8.

to read: "Report as the solidification point, to the nearest 0.01 C, the average of the highest readings, at least three, made at 10-sec intervals at the maximum level of temperature."

Tentative Method of Test for Water in Phenol and Related Materials by the Iodine Reagent Method (D 1631 – 59 T):[4]

Section 5 (c).—Change the first two sentences to read as follows:

(*c*) *Pyridine Solution*.—Place 4000 ml of refined grade pyridine in a 5000-ml distilling flask. Distill over and discard 400 ml of forecut at atmospheric pressure. Distill off 3400 ml of center cut and transfer this to a suitable glass bottle fitted with a two-hole stopper.

Note 1.—Change to read as follows:

NOTE 1.—The single solution reagent specified in the Method of Test for Water in Lacquer Solvents and Diluents (ASTM Designation: D 1364) may be used and is recommended if carbonyl compounds are suspected in the sample.

Section 6 (b).—To the third sentence add the words "to the nearest 0.1 mg."

Tentative Method of Test for Traces of Thiophene in Benzene Using Isatin and Spectrophotometry (D 1685 – 59 T):[4]

Section 3 (d).—Change to read: "(*d*) *Weighing Pipet*.—Any form is suitable. A convenient design is shown in Fig. 1." Delete footnote 4.

Section 6 (b).—Change the reference to Section 8 (*a*) to read "Section 8 (*b*)."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A on Monocyclic Aromatics (K. M. Brown, chairman) has been considering the problem of adding a Table IV, volume correction coefficients for mixed xylenes, to Method D 1555. This subcommittee is also engaged in editorially revising Methods D 848 and is investigating means of modifying the shaking procedure stipulated in that standard. The styrene task group has prepared methods for: inhibitor in styrene, polymer content of styrene monomer, solubility of styrene monomer, and aldehydes in styrene monomer, and has recommended that Method of Test for Kinematic Viscosity (D 445) and Method of Test for Color of Clear Liquids (Platinum-Cobalt Scale) (D-1209) be accepted by Committee D-16 as approved methods of test for styrene monomer.

Methods for traces of sulfur in aromatic hydrocarbons are also under study.

Subcommittee B on Polycyclic Aromatics (M. Mitchell, chairman) has been conducting cooperative tests of naphthalene solidification points using the National Bureau of Standards naphthalene thermometric standard. Task groups are also active on an acid wash test, sulfur in crude naphthalene, and crude naphthalene evaporation residue.

Subcommittee C on Phenolic Compounds (D. F. Pontz, chairman) has been actively studying methods for tar acids, phenol assay, and water solubility of phenol, and has drafted a proposed specification for refined phenol.

Subcommittee D on Nitrogen Heterocyclics (R. F. Shertzer, chairman) has discontinued work on refined quinoline due to lack of interest. Methods D 891, D 850, and D 1631 have been found acceptable for testing refined pyridine. Methods for color, water solubility, and oil content of refined pyridine will be considered.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee E on Thermometry (M. Lapeyrouse, chairman) has been active on problems related to the National Bureau of Standards' thermometric standards for phenol, naphthalene, and phthalic anhydride.

Subcommittee F on Editorial and Nomenclature (E. T. Scafe, chairman) has prepared drafts of an enlarged scope for Committee D-16 and a review of regulations governing technical committees. This subcommittee also functions as a consultant body to aid in the programs of the other subcommittees.

Subcommittee G on Statistical Procedure (W. J. Tancig, chairman) prepared the Recommendations for Interlaboratory Testing of Industrial Aromatic Hydrocarbons and Related Materials referred to earlier in this report. This subcommittee also serves as a consultant body to aid the other subcommittees when statistical problems are involved.

Subcommittee H, American Group ISO/TC 78, Aromatic Hydrocarbons (K. M. Brown, chairman).—The chairman, who was delegate to the third Plenary Meeting of ISO/TC 78 in Paris, Dec. 7 to 9, 1959, has presented a report on this meeting which covers several topics of great interest to Committee D-16. This subcommittee has also formulated comments for Committee D-16 on numerous documents originating in the ISO.

Subcommittee I on Phthalic Anhydride (O. E. Knapp, chairman) is planning cooperative testing of methods for solidification point, heat stability, and color of melt of phthalic anhydride.

This report has been submitted to letter ballot of the committee. which consists of 64 voting members; 54 members returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. E. SISCO,
Chairman.

K. H. FERBER,
Secretary.

APPENDIX

PROPOSED RECOMMENDATIONS FOR INTERLABORATORY TESTING OF INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS[1, 2]

These are proposed recommendations and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. These recommendations cover the general requirements for a cooperative interlaboratory testing program of an elementary type with reference to industrial aromatic hydrocarbons and related materials. It is to be used as a guide only and not as an inflexible framework. When unusual factors complicate the test, the recommendations should be modified as needed, and a practicing statistician, or a representative of ASTM Committee E-11 on Quality Control of Materials consulted. It is important to recognize that participation in a planned test program demands the statistical discipline imposed be observed by all cooperators. Anything short of absolute conformity to the procedures and imposed restrictions will be insufficient.

Summary

2. A simple routine is described for conducting an interlaboratory cooperative testing program. Arithmetical procedures are shown for eliminating data and calculating precision at the 95 per cent probability level. A suggested form for reporting the results of such a program is also included.

Definitions

3. (*a*) *Precision* is a measurement of the degree of agreement between replicate tests.

(*b*) *Replicate* means multiples of the same test on the same sample.

(*c*) *Accuracy* is a measurement of the difference between the average of the determined values and the true value.

(*d*) *Repeatability* is the precision of one operator using the same apparatus, expressed as the greatest difference between two results at the 95 per cent probability level.

(*e*) *Reproducibility* is the precision of a number of operators running the same test with different apparatus (Note), expressed as the greatest difference between two results at the 95 per cent probability level.

NOTE.—The "different" apparatus referred to is not different in design, only in identity.

(*f*) *Standard Deviation* (Note) is the square root of the sum of the squares of the deviations of the individual results from the group mean, divided by one less than the number of individual results. It may be obtained by subtracting the group mean or average from individual results, squaring these differences, adding the squares, dividing the sum of the squares by the number of individual values less one, and extracting the square

---

[1] These proposed recommendations are under the jurisdiction of the ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials.

[2] Published as information, December, 1960.

root of this value. The mathematical expression for this is:

$$s = \sqrt{\frac{(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \cdots + (X_n - \bar{X})^2}{n - 1}}$$

where:

s = estimated standard deviation,
$\bar{X}$ = test average,
$X_1, X_2, \cdots X_n$ = individual values, and
n = number of tests run.

Usually, it is preferable to use the following equation to determine the standard deviation:

$$s = \sqrt{\frac{n(\Sigma X^2) - (\Sigma X)^2}{n(n - 1)}}$$

This equation has the advantage that any error incurred in rounding off the mean does not enter into the answer. Such an effect is most apparent with very small numbers.

NOTE.—For simplicity, the estimated standard deviation, s, will be referred to as the standard deviation throughout this method.

(*g*) *Variance*, s^2, is the square of the standard deviation.

(*h*) *Degrees of Freedom* are the number of independent test conditions that can be varied. Generally this is $n - 1$.

Planning the Determination

4. (*a*) A planned evaluation of test results can be developed only by personnel who are intimately acquainted with all details of the test.

(*b*) The inclusion of *apparatus*, *operator*, and *laboratory* variables is necessary in every planned determination of precision, because ASTM methods are intended for general use. It must be assumed in such a determination that experienced operators and acceptable apparatus will be used. This reduces these variables to a minimum. Generally, the other variables that affect the test results will be of most importance in planning a determination of precision. An agreement must be reached by all cooperating laboratories as to which other significant variables should be included.

(*c*) Determine that all laboratories are prepared to run the test.

(*d*) The complete plan for a determination of precision should include the following:

(*1*) Names and addresses of participating laboratories, and number of operators each is to use.

(*2*) Specification of samples for each level in the range of expected test results, together with the number and source of each set.

(*3*) Test procedure to be followed in complete, agreed-upon detail.

(*4*) Identification of all essential apparatus and auxiliaries not included in Item (*3*).

(*5*) Complete definition of each variable to be considered, and the method and limits for its control during the determination.

(*6*) Any other applicable requirements, such as limitations on time between receipt of samples and their testing, elapsed time between start and end of testing, order of testing specimens, or method of reporting data.

(*7*) Statistical discipline to be established. The method shall be strictly adhered to, or data will be eliminated from the program.

General Requirements

5. (*a*) *Number of Participating Laboratories.*—It is desirable to have no fewer than six laboratories, with a maximum of ten, depending on the number of samples. In preliminary work, two or three laboratories will suffice.

(*b*) *Number of Samples.*—A minimum of three samples: one near each extreme of the concentration range or ranges intended to be covered by the test method, and one in the approximate middle of the range. More samples evenly spaced throughout the range are desired. For preliminary testing, one sample may be enough.

NOTE 1.—The gross sample must be homogeneous.

Procedure

6. (*a*) *Instructions.*—Issue detailed instructions to each participating laboratory in advance to include what is to be done with the samples, when it is to be done, how, and where the results are to be reported.

(*b*) *Samples.*—Withdraw enough portions from the gross sample or samples so that one portion may be sent to each participating laboratory. The gross sample should be large enough so that extra material will be available from the distributor in the event that any of the participating laboratories may need additional sample due to damaging of the original during shipment, etc.

(*c*) *Testing.*—Each laboratory shall conduct the specified number of tests on the samples according to instructions. A minimum of four tests shall be run on each sample at each laboratory; one operator shall test each sample in duplicate on each of two consecutive days.

NOTE 2.—Any test results suspected because of improvisation, departure from test instructions, or other unusual circumstances during the test shall be automatically excluded from the calculations and report.

Calculation

7. (*a*) Examine the data for completeness.

(*b*) Eliminate any results known to have been determined incorrectly.

(*c*) Test and eliminate any suspect data (see Appendix).

(*d*) Calculate the repeatability of the method as follows: When k operators are involved, if Operator 1 makes n_1 determinations of which the mean is $\bar{X}_1$ with any individual result X_1, Operator 2 makes n_2 determinations of which the mean is $\bar{X}_2$ with any individual result X_2, and so on to Operator k, obtain the pooled estimate of the repeatability standard deviation S_r, from the following equation:

$$S_r = \sqrt{\frac{\Sigma(X_1 - \bar{X}_1)^2 + \Sigma(X_2 - \bar{X}_2)^2 + \cdots + \Sigma(X_k - \bar{X}_k)^2}{(n_1 - 1) + (n_2 - 1) + \cdots + (n_k - 1)}}$$

Multiply the S_r thus calculated by the appropriate multiplier from Table I corresponding to $(k - 1)$ degrees of freedom. This value is the repeatability of the method.

TABLE I.—MULTIPLIERS OF THE ESTIMATE OF THE STANDARD DEVIATION FOR CALCULATING THE GREATEST DIFFERENCE BETWEEN TWO RESULTS AT THE 95 PER CENT PROBABILITY LEVEL.

Degrees of Freedom	Multiplier	Degrees of Freedom	Multiplier	Degrees of Freedom	Multiplier
1	17.97	13	3.05	25	2.91
2	6.08	14	3.03	26	2.91
3	4.50	15	3.01	27	2.90
4	3.92	16	3.00	28	2.90
5	3.64	17	2.98	29	2.89
6	3.46	18	2.97	30	2.89
7	3.34	19	2.96	40	2.86
8	3.26	20	2.95	50	2.84
9	3.20	21	2.94	60	2.83
10	3.15	22	2.93	120	2.80
11	3.11	23	2.93	∞	2.77
12	3.08	24	2.92		

(*e*) Calculate the reproducibility of the method as follows: When k operators, each in different laboratories, produce results the average of which is $\bar{X}$ and the grand average is $\bar{\bar{X}}$, then calculate the reproducibility standard deviation, S_R, from the following equation:

$$S_R = \sqrt{\frac{(\bar{X}_1 - \bar{\bar{X}})^2 + (\bar{X}_2 - \bar{\bar{X}})^2 + \cdots + (\bar{X}_k - \bar{\bar{X}})^2}{(k - 1)}}$$

Multiply the S_R thus calculated by the appropriate factor from Table I corresponding to $(k - 1)$ degrees of freedom. The value obtained is the reproducibility of the method.

Reporting

8. (*a*) The following data should be used for judging the acceptability of results (95 per cent probability), and should be included in the conclusion of any method:

(*1*) Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

Range or Sample Description	Repeatability	Number of Cooperating Laboratories	Total Number of Individual Tests
....	....	....	....
....	....	....	....

(*2*) The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts:

Range or Sample Description	Reproducibility	Number of Cooperating Laboratories	Total Number of Operators
....	....	....	....
....	....	....	....

(*3*) The first year the method is published it is recommended that all of the cooperative data used to determine Items (*1*) and (*2*) be presented in tabular form. During subsequent years a note should be included to indicate the year the data were published.

(*b*) *Submitting Methods.*—When submitting to the Society a method for which precision has been determined by this program, include a statement to that effect in the accompanying report of the committee having jurisdiction. The plan of the determination and summary of the data obtained should be appended to the report.

APPENDIX

TESTING SUSPECT DATA

A1. When extreme values are suspected of being outside the normal range of cooperative test data from one laboratory, they should be tested by applying the following test (Dixon's "r" test):

Arrange the data in ascending order, such that $X_1 \leq X_2 \leq X_3 \cdots \leq X_n$, and calculate the r value, using the proper equation of Table II. If this r value is larger than the corresponding value in Table II, then the suspect datum should be rejected.

Example 1:

Five test observations are obtained as follows:

22.1, 22.7, 22.9, 23.1, and 25.6

Step 1.—Arrange the values in ascending order:

X_1	X_2	X_3	X_4	X_5
22.1	22.7	22.9	23.1	25.6

Step 2.—Since the number of samples is five, and since the suspected value is the largest in the set, calculate the r value as follows:

$$r = \frac{X_5 - X_4}{X_5 - X_1} = \frac{25.6 - 23.1}{25.6 - 22.1} = \frac{2.5}{3.5} = 0.71$$

Step 3.—Since the r value is larger than the value of 0.642 in Table II at the 95 per cent level of significance, the value 25.6 should be discarded.

Example 2:

Eleven test observations are obtained as follows:

3.17, 3.21, 2.98, 3.07, 3.13, 3.06, 2.77, 2.97, 2.99, 3.14, 3.12

Step 1.—Arrange the values in ascending order:

X_1	X_2	X_3	X_4	X_5	X_6
2.77	2.97	2.98	2.99	3.06	3.07

X_7	X_8	X_9	X_{10}	X_{11}
3.12	3.13	3.14	3.17	3.21

Step 2.—Since the number of samples is

TABLE II.—TESTING FOR EXTREME OBSERVATION.[a]
(No Past Data)

n	Criterion for *r* Value	Critical Value at 95 per cent Level
3	$r = \frac{X_2 - X_1}{X_n - X_1}$ if *smallest* value is suspect $r = \frac{X_n - X_{n-1}}{X_n - X_1}$ if *largest* value is suspect	0.941
4		0.765
5		0.642
6		0.560
7		0.507
8	$r = \frac{X_2 - X_1}{X_{n-1} - X_1}$ for *smallest* value $r = \frac{X_n - X_{n-1}}{X_n - X_2}$ for *largest* value	0.554
9		0.512
10		0.477
11	$r = \frac{X_3 - X_1}{X_{n-1} - X_1}$ for *smallest* value $r = \frac{X_n - X_{n-2}}{X_n - X_2}$ for *largest* value	0.576
12		0.546
13		0.521
14	$r = \frac{X_3 - X_1}{X_{n-2} - X_1}$ for *smallest* value $r = \frac{X_n - X_{n-3}}{X_n - X_3}$ for *largest* value	0.546
15		0.525
16		0.507
17		0.490
18		0.475
19		0.462
20		0.450
21		0.440
22		0.430
23		0.421
24		0.413
25		0.406

---

[a] By permission from "Introduction to Statistical Analysis" by W. J. Dixon and F. J. Massey' Jr.

Copyright 1957 (2nd Ed.), McGraw-Hill Book Co., Inc.

eleven, and since the suspected value is the smallest in the set, calculate the *r* value as follows:

$$r = \frac{X_3 - X_1}{X_{n-1} - X_1} = \frac{2.98 - 2.77}{3.17 - 2.77} = \frac{0.21}{0.40} = 0.525$$

Step 3.—Since the *r* value is smaller than the value of 0.576 in Table II at the 95 per cent level of significance, the value 2.77 cannot be discarded.

NOTE.—If any of the remaining data still appear to be out of line, apply the "*r*" test as often as necessary, or until the test indicates no more data may be eliminated.

Example 3:

For the following check values test the most divergent for rejection:

3.30, 3.44, 3.50, 3.51, 3.52, 3.53

(*1*) At the 95 per cent level, *r* value for 3.30 = 0.61 versus value of 0.56 in Table II; therefore, reject.

(*2*) At the 95 per cent level, *r* value for 3.44 = 0.67, versus value of 0.64 in Table II; therefore, reject.

(*3*) At the 95 per cent level, *r* value for 3.50 = 0.33 versus value of 0.765 in Table II; therefore, do not reject.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES*

Committee D-17 on Naval Stores held one meeting during the year: on June 25, 1959, at Atlantic City, N. J., in connection with the Annual Meeting of the Society.

The committee consists of 30 members, of whom 12 are classified as producers, 7 as consumers, and 11 as general interest members.

The following changes in personnel occurred during the year: Douglas E. Campbell was appointed to Committee D-17 as a representative of the Pulp Chemicals Assn., Tall Oil Division. Neuman R. Craig was appointed a new member from the U. S. Department of Agriculture. W. D. Pohle, of Swift and Co., resigned.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. R. Snider.
Vice-Chairman, J. L. Boyer.
Secretary, W. A. Kirklin.

American Standard

The following standard under the jurisdiction of Committee D-17 has been approved as American Standard by the American Standards Association:

Standard Methods of:

Sampling and Testing Turpentine (ASTM D 233 - 58; ASA K 33.1—1959).

Activities of Subcommittees

Subcommittee I on Softening Point of Rosin (J. N. Borglin, chairman) completed cooperative studies in eight laboratories on the softening point of several rosins using the Newport capillary tube method, the Hercules thermometer drop method, and the ASTM ring and ball method (E 28 - 58 T). Four rosin type products were used, namely, FF wood, tall oil, gum, and a higher melting modified wood rosin. Results showed the ring and ball method to be equally if not more reproducible than either of the other two methods, and to be preferred for referee purposes. The replies to a questionnaire on the subject were such as to question further testing of an alternate method at this time.

Subcommittee IV on Chemical Analysis of Rosin (R. Herrlinger, chairman) is planning revisions in Methods of Test for Fatty Acids Content of Tall Oil Rosin (D 1585 - 58 T).

Subcommittee V on Tall Oil (J. J. Owen, chairman) is refining the current Methods of Testing Tall Oil (D 803 - 58), including viscosity and color and is working on revisions of Method of Test for Rosin Acids in Fatty Acids (D 1240 - 54). This work reflects the growing utilization of tall oil in the naval stores industry.

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman) has recommended that the description of the distillation apparatus in the distillation tests in Methods D 233 for turpentine, D 801 for dipentene, and D 802 for pine oil be deleted and that these tests make reference to Standard Specifications for Distillation Equipment (E 133 – 58). It is also recommended that reference to Methods of Test for Distillation of Gasoline, Naphthalene, and Similar Petroleum Products (D 86 – 59) be deleted. Use of the 200-ml distillation flask in Specifications E 133 has been questioned and use of only the 100-ml flask will be continued for turpentine, dipentene, and pine oil (and other terpene products) pending further study.

---

* Sixty-third Annual Meeting of the Society, June 26-July 1, 1960.

Subcommittee IX on Definitions (J. L. Boyer, chairman) is cooperating with the Tall Oil Division of the Pulp Chemicals Assn. on definitions for tall oil products.

This report has been submitted to letter ballot of the committee, which consists of 28 members; 18 members returned their ballots, of whom 16 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. R. Snider,
Chairman.

W. A. Kirklin,
Secretary.

REPORT OF COMMITTEE D-18
ON
SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held two meetings during the year: in Atlantic City, N. J., on June 24, 1959, and in San Francisco, Calif., on Oct. 15, 1959. Executive Committee meetings were held on June 22 and 23, 1959, in Atlantic City, and in Washington, D. C., on January 10, 1960. A joint meeting of the Executive Committees of Committee D-4 on Road and Paving Materials and Committee D-18 was held on June 23, 1959, at Atlantic City.

During the Annual Meeting of the Society in Atlantic City, the following papers sessions were held: (1) Symposium on Time Rates of Loading and Soil Testing, (2) Symposium on Atterberg Limits, and (3) a Session on Application of Soil Testing in Highway Design and Construction. A two-part general soils session was held during the San Francisco meeting of the Society at which time nine papers were presented. The symposia and papers will be published by the Society as a special technical publication *ASTM STP No. 254.*

Also included in the activities at the San Francisco meeting was a tour of three large soil testing laboratories in the Bay area.

Resignations were accepted during the year from J. R. Schuyler, New Jersey State Highway Department; L. M. Pellow, Solvay Process Division, Allied Chemical Corp.; L. A. Palmer, U. S. Navy Bureau of Yards and Docks; J. M. Griffith, Asphalt Inst.; and D. P. Warner, Karol-Warner.

The committee consists of 144 members, of whom 110 are voting members; 22 are classified as producers, 39 as consumers, and 49 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. G. Holtz.

First Vice-Chairman, C. B. Crawford.

Second Vice-Chairman, W. G. Shockley.

Secretary, A. A. Wagner.

Membership Secretary, J. P. Gnaedinger.

Special Committee for Hogentogler Award.—This special committee, consisting of L. E. Gregg (chairman), D. M. Burmister, and W. E. Schmidt, recommended that the Hogentogler Award be given to Robert L. Schiffman and Charles R. Wilson for their paper entitled "The Mechanical Behavior of Chemically Treated Granular Soils," presented at the 1958 Annual Meeting.

Symposia Committee.—H. F. Clemmer was appointed to head a committee for a Symposium on Nuclear Devices for Measuring Soil Moisture and Density[1] to be held at the approaching Annual Meeting. This symposium will be of the panel discussion type, with all papers being made available prior to the meeting.

Committee G-2 on Special Papers.—Philip Keene (chairman), W. T. Spencer, and C. R. Foster were appointed to secure papers for a general soils session to be held during the Annual Meeting.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] To be published as *ASTM STP No. 293.*

Joint D-4 - D-18 Subcommittee.—At a joint meeting of the executive subcommittees of Committees D-4 and D-18 it was agreed that the joint subcommittee be abolished. It was further agreed that the activities previously handled by the joint subcommittee would be handled by a new Subcommittee S-8 on Special and Construction Control Specifications, of Committee D-18. Committee D-4 will have official representation on this new subcommittee.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1959 Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Definitions of:

Terms Relating to Soil Dynamics.

Revision of Tentative Method for:

Grain-Size Analysis of Soils **(D 422 - 54 T)**.

Revision of Tentative Method of Test for:

Liquid Limit of Soils **(D 423 - 54 T)**.

The revisions of Tentative Methods D 422 and D 423 were accepted by the Standards Committee on Nov. 9, 1959, and they appear in the 1959 Supplement to Book of ASTM Standards, Part 4. The proposed Tentative Definitions of Terms Relating to Soil Dynamics are still before the Standards Committee.[2]

Revision of Tentative

The committee recommends revisions as follows of the Tentative Method for Grain-Size Analysis of Soils (**D 422–59 T**)[3] and continuation of the method as tentative:

Section 2(f).—Add 3-in. to the list of sieves.

Section 4(a).—To the tabulation in Item (*1*) add an additional particle size with a nominal diameter of 3 in. and an approximate minimum weight of 5000 g.

Section 5(a).—Add 3-in. to the list of sieves.

Section 17.—Change Paragraph (*c*) to read as follows:

> (*c*) For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 sieve, the results read from the graph may be reported as follows:
>
> (*1*) Gravel, passing 3-in. and retained on No. 4 sieve................ .... per cent
> (*2*) Sand, passing No. 4 sieve and retained on No. 200 sieve............ .... per cent
>   (*a*) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve............ .... per cent
>   (*b*) Medium sand, passing No. 10 sieve and retained on No. 40 sieve...... .... per cent
>   (*c*) Fine sand, passing No. 40 sieve and retained on No. 200 sieve......... .... per cent
> (*3*) Silt size, 0.074 to 0.005 mm................. .... per cent
> (*4*) Clay size, smaller than 0.005 mm........... .... per cent
>   (*a*) Colloids, smaller than 0.001 mm............ .... per cent

In Paragraph (*d*) in the fourth line change "No. 10" to read "No. 4" and add 3-in. to the list of sieves in the tabular material.

Adoption of Tentative as Standard Without Revision

The committee recommends that the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (**D 653 –**

---

[2] The tentative definitions were accepted by the Standards Committee on September 22, 1960, and appear in the 1960 Supplement to Book of ASTM Standards, Part 4, bearing the designation D 1707 – 60 T.

[3] 1959 Supplement to Book of ASTM Standards, Part 4.

58 T)[4] be approved for reference to letter ballot of the Society for adoption as standard without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Test for Moisture-Density Relations of Soils, Using 5.5-lb Rammer and 12-in. Drop (**D 698 – 58 T**),

Testing Soil-Bituminous Mixtures (**D 915 – 47 T**),

Test for Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (**D 916 – 47 T**),

Test for Load-Settlement Relationship for Individual Piles Under Vertical Axial Load (**D 1143 – 57 T**),

Test for Water-Soluble Chlorides Present as Admixtures in Graded Aggregate Road Mixtures (**D 1411 – 56 T**),

Test for Soil Investigation and Sampling by Auger Borings (**D 1452 – 56 T**),

Test for Density of Soil in Place by the Sand-Cone Method (**D 1556 – 58 T**),

Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils (**D 1558 – 58 T**),

Test for Moisture-Density Relations of Soils, Using 10-lb Rammer and 18-in. Drop (**D 1557 – 58 T**),

Penetration Test and Split-Barrel Sampling of Soils (**D 1586 – 58 T**), and

Thin-Walled Tube Sampling of Soils (**D 1587 – 58 T**).

Tentative Specifications for:

Materials for Soil Aggregate Subbase, Base, and Surface Courses (**D 1241 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee G-1, Editorial (E. E. Bauer, chairman).—The chairman has checked many details of an editorial nature in connection with the various tentatives and standards under the jurisdiction of Committee D-18.

Subcommittee G-3, Nomenclature and Definitions (C. R. Foster, chairman) recommended the adoption of Definitions D 653 - 58 T as standard; approved the compilation of a single joint publication by the ASTM and the ASCE of the definitions accepted by the two cooperating committees; and instituted arrangements with the ASCE for a single joint committee which can more efficiently conduct future cooperative work. Suggestions for needed work and means of expediting committee work are being formulated for subcommittee action.

Steering Committee on Research (W. S. Housel, chairman) held a meeting during the Annual Meeting of the Society. The chairman of the Steering Committee attended most of the subcommittee meetings held during the Annual Meeting in order to present the objectives of the Steering Committee, to complete the list of unsolved problems, and to discuss the status of research in each subcommittee's field. Subcommittees contacted were R-2, R-4, R-5, R-8, R-9, and R-11.

The Steering Committee also endorsed the proposal to establish a headquarters repository for committee research reports, and recommended to Committee D-18 that this proposal be called to the attention of the R-subcommittees as a procedure to preserve research data and unpublished reports that may otherwise be lost.

Subcommittee R-3, Physical Characteristics of Soils (E. E. Bauer, chairman) is recommending certain changes in Method D 422, as noted earlier in the report, in order to eliminate conflict with Definitions D 653. Consideration is being given to the stirring paddle illustrated in Fig. 1 of Method D 422.

The subcommittee recommends the continuation as tentative of Methods D

---

[4] 1958 Book of ASTM Standards, Part 4.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

698, D 1557, and D 1558 during consideration of future revisions.

A paper by Raymond F. Dawson entitled "Some Laboratory Studies of the Moisture-Density Relations of Soils," presented at the San Francisco Meeting in October 1959, gave test results comparing sector heads with circular heads; 4- and 6-in. molds; and hand tamping with machine tamping.[6]

Attention is being given to the preparation of methods of test for the determination of maximum and minimum densities of soils for the determination of relative density, and to the inclusion of a tolerance provision of plus or minus 5 C (9 F) whenever a drying temperature is stated in any method of test under the jurisdiction of the subcommittee.

Subcommittee R-4, Physical Properties of Soils (N. W. McLeod, chairman) is drafting methods for measuring the permeability and capillarity of soils. The method under consideration for capillarity is being restricted to soils for which the capillary potential is less than one atmosphere.

Subcommittee R-6, Physico-Chemical Properties of Soils (D. T. Davidson, chairman) has continued to work with the American Society of Agronomy responsible for the preparation of a Monograph, "Methods of Soil Analysis," reviewing the outline, offering suggestions, and arranging with ASTM members to write certain sections in the Monograph. In December 1959, the Board of Directors of the ASA voted to invite the ASTM to act as co-sponsor of the Monograph, to the extent that the Monograph would bear the imprint of both societies and would give credit to the committees and members of the two societies for preparation of the work. There would be no financial obligation involved on the part of the ASTM in such co-sponsorship. They voted further that closer liaison involving joint responsibility for the costs of publication and sharing of the profit or loss should be cleared through the Monographs Committee of the ASA. The Board of Directors of ASTM voted to accept this invitation to act as co-sponsor of the Monograph without financial obligation, subject to concurrence by Committee D-18.

Plans for the coming year include: (1) further cooperation with the ASA in the preparation of the Monograph, and (2) an appraisal of the need for and desirability of developing standard methods of testing physicochemical properties of soils.

Subcommittee R-8, Special and Construction Control Tests (H. F. Clemmer, chairman).—Section A, Base Courses and Subgrades for Highways and Airports, has prepared a method of test for the bearing ratio of soils (CBR) and will study the swelling properties of soils and the promotion of other methods for bearing tests. Section B, Earth Dams and Embankments, has submitted a proposed method of test for determining the density of soil in-place by the rubber balloon method. The section proposes to study other methods of determining density and is interested in studying the nuclear method for determining moisture and density.

Section C-1, Cement Stabilization, is interested in a new method of testing soil-cement for cement content. Section C-2, Bituminous Stabilization, has recommended the adoption as standard of Specifications D 915, with an editorial revision of the scope, and is considering a specification for soil-bituminous stabilization. Section C-3, Calcium Chloride, is preparing revisions to ASTM Specifications D 1241 to conform with AASHO M-147.

The subcommittee recommends the continuation as tentative of Specification D 1241 and Methods D 915, D 916, D 1411, and D 1556 pending consideration by the sectional committees for revision.

---

[6] This paper is included in *ASTM STP No. 254*.

Subcommittee R-9, Dynamic Properties of Soils (R. K. Bernhard, chairman).—The proposed Tentative Definitions of Terms Relating to Soil Dynamics has been submitted to the Administrative Committee on Standards for adoption as tentative.[2]

At a subcommittee meeting on June 23, 1959, the advantages and difficulties in measuring and computing shear-stresses or strains of soils *in situ* subjected to dynamic loads were outlined by Mr. R. G. Ahlvin. In connection with the successful application of periodic loads, a paper on "Experiments with Vibratory Driving of Piles" by Mr. M. M. Levkin, Chief Engineer of Large Bridges, U.S.S.R, was discussed. Some results illustrated by lantern slides on recent investigations referring to two- and three-dimensional motions of soils *in situ* excited by periodic and nonperiodic forces, as well as discrimination between impact (explosive-subsurface nuclear blasts) and earthquake effects, were presented by the chairman.

Subcommittee R-11, Pile Load Bearing Tests (W. S. Housel, chairman) is recommending that Methods D 1143 be continued as tentative. Four unsolved problems needing research have been presented to the subcommittee and action is pending to solicit cooperative research projects on these problems. The subcommittee is also studying a suggested method of test for the lateral load capacity of batter piles or pile frames with the objective of submitting a tentative method.

This report has been submitted to letter ballot of the committee, which consists of 110 voting members; 90½ members returned their ballots, of whom 89½ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Chairman.

W. G. HOLTZ,
Secretary.

REPORT OF COMMITTEE D-19
ON
INDUSTRIAL WATER*

Committee D-19 on Industrial Water held two meetings during the year: on June 24–26, 1959, in Atlantic City, N. J., and on Jan. 25–27, 1960, in Atlanta, Ga.

The Symposium on the Identification of Water-Formed Deposits, sponsored by the committee and presented on June 25, 1959, has been published as *ASTM STP No. 256*. The Symposium on Technical Developments in the Handling and Utilization of Water and Industrial Waste Water, presented during the Third National Pacific Area National Meeting, will be published as *ASTM STP No. 273*.

"Old Wine in New Bottles—A New Look at the Substance, Water" was the title of a Luncheon talk during the West Coast Meeting by F. E. Clarke, U.S. Navy Engineering Experiment Station, Annapolis, Md. This address has been published in the ASTM BULLETIN, No. 243, Jan. 1960, p. 17.

The Manual on Industrial Water and Industrial Waste Water, Second Edition, was published in 1959 as *ASTM STP No. 148-D*.

The Administrative Committee on Research approved the research project "Investigation of Vaporous Carry-over of Boiler Water Salts," and granted Committee D-19 $6000 to defray the cost of the investigation. A contract to perform the investigation was awarded by Committee D-19 to the Research Center of the Babcock & Wilcox Co., Alliance, Ohio. The investigation is under way and is expected to be completed before the end of 1960.

The 1960 Max Hecht Award certificate and citation will be presented to F. N. Speller on June 30, 1960.

The ASTM representatives serving on the Joint Committee of Uniformity of Methods of Water Examination (JCUMWE) submitted to the Society and the committee recommendations relating to Committee D-19 methods as follows, covering the period of July 1, 1959 to July 1, 1960:

Preliminary recommendations were submitted by JCUMWE relative to the following: acidity, basicity, and alkalinity; dissolved oxygen; manganese; pH; sulfate; and uniformity of reagents. Committee D-19 suggested reconsideration of the recommendations on dissolved oxygen and, in conjunction with Committee E-1 on Methods of Testing, on pH. The committee offered no comment on the recommendation on manganese. Committee comments on the balance of the preliminary recommendations are pending.

Official recommendations were submitted by JCUMWE relative to the following: grease and oily matter; hardness; iron; organic nitrogen; and suspended and dissolved matter (solids). Committee D-19 adopted all the recommendations except the last named. Action on this recommendation is pending.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

JCUMWE established new review panels to consider ammonia; calcium and magnesium; carbon dioxide, bicarbonate, and carbonate; electrical conductivity; fluoride; lead; nitrite; total phosphorus; and turbidity. ASTM will be represented on all these panels except that on lead.

Committee D-19 is operating with one advisory and eight technical subcommittees, effective June 30, 1960. The committee accepted changes of the titles and scopes of Subcommittees III, IV, V, VI, and VII as well as the title and scope of the newly established Subcommittee VIII and an editorial revision in the scope of Committee D-19. The titles and scopes of all the subcommittees are as follows:

Committee D-19:

Scope: Delete the words "water-formed" before "deposits."

Subcommittee I:

Title: Advisory (Administrative).

Subcommittee II:

Title: Definitions and General Specifications.

Scope: The scope of this subcommittee comprises the preparation for publication purposes of special material that is not a method of test or of sampling but is of such a general nature as to fall outside the scope of any other subdivision of the committee. Nomenclature, terminology, and general specifications shall be considered as special material.

Subcommittee III:

Title: Methods of Sampling and Flow Measurements.

Scope: This committee shall prepare methods for the sampling and flow measurement of industrial water in its various states and of industrial waste water. It shall also prepare methods for the sampling of deposits resulting from or incident to the industrial use of water and specifications for equipment for the sampling and flow measurements.

Subcommittee IV:

Title: Methods of Analysis of Industrial Water.

Scope: This subcommittee shall prepare methods for physical or chemical testing of industrial water where the specialized nature of the apparatus required is not controlling.

Subcommittee V:

Title: Methods of Instrumental Analysis.

Scope: This subcommittee shall prepare methods for physical or chemical testing of water and water-formed deposits wherein the specialized nature of the apparatus required is controlling.

Subcommittee VI:

Title: Performance Testing.

Scope: This subcommittee shall prepare methods for evaluating the performance (including corrosivity) of all types of industrial water and industrial waste water under various conditions of industrial use.

Subcommittee VII:

Title: Methods of Analysis of Industrial Waste Water.

Scope: This subcommittee shall prepare methods for physical, chemical, or biological testing of industrial waste water where the specialized nature of the apparatus required is not controlling.

Subcommittee VIII:

Title: Methods of Analysis of Deposits.

Scope: This subcommittee shall prepare methods for physical or chemical testing of deposits, including corrosion products, resulting from or incident to the industrial use of water where the specialized nature of the apparatus required is not controlling.

Subcommittee IX:

Title: Methods of Radiochemical Analysis.

Scope: This subcommittee shall prepare methods for physical and chemical testing of industrial water, industrial waste water, and deposits where radioactivity or radiochemical considerations are controlling.

Subcommittee IX on Radiochemical Analysis was established on June 30, 1960.

The committee comprises 144 members. The committee operates with 67 task groups divided among eight technical subcommittees. The committee's production of methods includes 36 tentatives accepted and published by the Society and 35 Standards adopted and published by the Society. The tentatives and standards included in this report are excluded from the totals.

The officers elected for the ensuing term of two years are as follows:

Chairman, Max Hecht.

Vice-Chairman, F. E. Clarke and F. R. Owens.

Secretary, O. M. Elliot.

Standards Advisor (appointed), C. K. Rice.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the recommendation for the revision of Tentative Method of Test for Odor in Industrial Water and Industrial Waste Water (D 1292 - 58 T). This recommendation was accepted by the Standards Committee on Oct. 2, 1959, and the method appears in the Manual on Industrial Water and Industrial Waste Water, Second Edition, 1959.

NEW TENTATIVES

The committee recommends for publication as tentative the foilowing methods as appended hereto:[1]

Tentative Method of Test for:

Operating Performance of Cation Exchange Materials—Sodium Cycle, and

Phenolic Compounds in Industrial Water and Industrial Waste Water.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Test for Phosphate in Industrial Water (D 515 - 58 T):[2] Revise as appended hereto.[1]

Tentative Methods of Test for Iron in Industrial Water and Industrial Waste Water (D 1068 - 55 T):[2] Revise as appended hereto.[1]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision in substance. Two of the methods are to be revised editorially, as indicated later in the report.

Tentative Method of Test for:

Identification of Types of Microorganisms and Microscopic Matter in Industrial Water and Industrial Waste Water (**D 1128 - 58 T**),[2]

Nitrite Ion in Industrial Water (**D 1254 - 57 T**),[2]

Specific Gravity of Industrial Water and Industrial Waste Water (**D 1429 - 56 T**),[2]

Dissolved and Gaseous Hydrogen in Industrial Water (**D 1588 - 58 T**),[2]

Dissolved Oxygen in Industrial Waste Water (**D 1589 - 58 T**),[2] and

Surface Tension of Industrial Water and Industrial Waste Water (**D 1590 - 58 T**).[2]

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Method of Test for Chloroform-Extractable Matter in Industrial Water and Industrial Waste Water (D 1178 - 54 T):[2]

New Footnote.—Add a new Footnote *3a* against the word "matter" in Section 1(*a*) to read as follows:

---

[1] The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 10.

[2] 1958 Book of ASTM Standards, Part 10.

[3a] The American Society for Testing Materials agrees with the conclusion reached in November, 1959, by the Joint Committee on Uniformity of Methods of Water Examination, which states that uniformity of methods for the determination of grease and oily matter is not practical on the basis of present technical knowledge.

New Note.—At the end of Section 1(*b*) place the following new Note 1, renumbering subsequent Notes accordingly:

NOTE 1.—The Joint Committee on Uniformity of Methods of Water Examination has concluded that uniformity of methods for the determination of grease and oily matter is not practical on the basis of present technical knowledge.

Commonly used solvents are hexane, petroleum ether, benzene, chloroform, or carbon tetrachloride. These solvents exert selective extraction of specific greases and oily constituents. In addition, nonoily materials, such as phenolic type material and colloidal sulfur, are selectively extracted to varying degrees by these solvents. The selectivity of extraction is affected by the sample-to-solvent ratio.

Oily matter and grease may be of mineral, animal, or vegetable origin. The solvent action exerted on material of such different chemical structure will vary to a marked degree. Thus, application of a test method for oily matter or grease to such materials will necessarily produce a variety of results depending on the solvent used. In one case, a solvent may be an excellent extractant of mineral oil and a poor extractant of vegetable oil. In another case, a second solvent may be a poor extractant for mineral oil but excellent for extracting vegetable oil.

The definition of grease and oily matter by necessity is based on the procedure used because of the above considerations. The source of the grease or oily matter, the solvent used, the sample-to-solvent ratio, the pH of the sample, and the inclusion of nonoily matter will dictate the material determined and influence the interpretation of the results obtained.

Tentative Method of Test for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Industrial Waste Water (D 1252 – 58 T):[2]

Section 8.—Add the following sentence at the end of Paragraph (*d*): "If such compounds are present (especially high chloride concentration), modify the procedure as described in Paragraph (*g*)."

Add the following as Paragraph (*g*) and new Note 5:

(*g*) When high chloride concentrations are present, reflux the mixture for 1 hr in the absence of the Ag_2SO_4 catalyst. This will volatilize the chloride as free chlorine. Stop the refluxing, allow to cool, and then add 1 g of Ag_2SO_4 (Note 5). Continue refluxing for 1 hr, then proceed as described in Paragraph (*b*).

NOTE 5.—A special flask carrying a side arm to facilitate the introduction of the Ag_2SO_4 is preferable. The catalyst may also be made in pellet form, each pellet containing about 1 g of Ag_2SO_4. This makes introduction of the catalyst into the reflux mixture easier.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods of Test for Hardness in Industrial Water (D 1126 – 57):[2]

Footnote 2.—Add a paragraph to read as follows: "This method conforms essentially to the recommendations made by the Joint Committee on Uniformity of Methods of Water Examination in March, 1959."

Note 1.—Change to read as follows:

NOTE 1.—Hardness is also caused by other polyvalent cations which seldom are present in more than trace amounts. Strontium will be titrated with calcium in both the referee and non-referee methods. The interfering effects of other cations such as aluminum, barium, iron, manganese, and zinc may be eliminated by addition of appropriate inhibitors.

Section 14.—Rearrange paragraphs so that reagents are listed in alphabetical sequence. Change Paragraphs (*a*) and (*b*) to read as follows:

(*a*) *Ammonium Hydroxide Solution* (*1:4*).—

Mix 1 volume of NH_4OH (sp gr 0.90) with 4 volumes of water.

(*b*) *Hydrochloric Acid Solution* (*1:4*).—Mix 1 volume of HCl (sp gr 1.18) with 4 volumes of water.

Change Item (*3*) of Paragraph (*c*) to read as follows:

(*3*) When cool, mix the two solutions and add 1 g of magnesium ethylenediamine tetraacetate salt, having a ratio of 1 Mg:1 EDTA. Complex any excess magnesium with standard sodium ethylenediamine tetraacetate solution (1 ml = 1.0 mg $CaCO_3$) if necessary. Make up to 1 liter with water. Keep the solution bottle stoppered when not in use. The reagent will be effective for at least one month.

Change Paragraph (*f*) to read as follows: "(*f*) *Sodium Hydroxide Solution* (*50 g per liter*).—Dissolve 50 g of NaOH in water and dilute to 1000 ml."

Sections 18 to 23.—Delete these sections, which cover the present Non-Referee Method B.

Standard Definitions of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 58):[2]

New Note.—To the definitions for Backwash, Ion Exchange, Ion-Exchange Capacity, Ion-Exchange Material, Operating Cycle, Regeneration, Regeneration Level, Rinse, Service Run, and Sodium Cycle add a Note to read: "NOTE.—This definition applies to solid-phase ion-exchange materials."

In the definition of Ion-Exchange Capacity replace "which" with the word "that." In the definition of Sodium Cycle replace the words "removal of specified ions" with the words "removal of specified cations."

Revise existing definitions to read as follows:

Acidity.—The quantitative capacity of aqueous media to react with hydroxyl ions. (D 1067)

Alkalinity.—The quantitative capacity of aqueous media to react with hydrogen ions. (D 1067) (See *Basicity.*)

Basicity.—The quantitative capacity of aqueous media to react with hydrogen ions. (D 1067) (See *Alkalinity.*)

Turbidity.—Reduction of transparency of a sample due to the presence of particulate matter.

New Definitions.—Add the following new definitions in proper alphabetical sequence:

Background.—In the measurement of radioactivity of water, the counting rate observed when the sample is replaced by an equal volume of reagent water. Examples of potential background include cosmic rays, contaminating radioactivity, and electrical noise. (D 1690)

Counter Efficiency.—In the measurement of radioactivity, that fraction of the disintegrations occurring in a source which is detected by the counter. (D 1690)

Counter Gamma Efficiency.—In the measurement of radioactivity, that fraction of gamma photons emitted by a source which is detected by the counter. (D 1690)

Curie.—A unit of radioactivity equivalent to 3.7×10^{10} atomic disintegrations per second or 2.22×10^{12} atomic disintegrations per minute. A microcurie is 1 one-millionth of a curie (10^{-6} curie); a micromicrocurie or picocurie, 1 one-millionth of a microcurie (10^{-12} curie). (D 1690)

Diatoms.—Microscopic unicellular or colonial algae constituting the class Bacillarieae and having silicified cell walls.

Inhibitory Toxicity.—Any demonstrable direct inhibitory action of a substance on the rate of general metabolism (including rate of reproduction) of living organisms.

Odor Intensity Index.—The number of times the concentration of the original sample is halved by addition of odor-free water to obtain the least definitely perceptible odor. (D 1292)

Radioactively Homogeneous Water.—Water in which the radioactivity is uniformly dispersed throughout the volume of water and remains so dispersed throughout the course of the measurement. (D 1690)

Threshold Odor Number.—The greatest dilution of the sample with odor-free water to give the least definitely perceptible odor. (D 1292)

Standard Method of Test for Oily Matter in Industrial Waste Water (D 1340 - 56):[2]

New Footnote.—Add a new Footnote *3a* against the word "matter" in Section 1(*a*) to read as shown above for Method D 1178.

New Note.—At the end of Section 1 place a new Note 1 to read as shown above for Section 1(*b*) of Methods D 1178, renumbering subsequent Notes accordingly.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision in substance of the following tentatives:

Tentative Methods of:

Sampling Industrial Water (**D 510 - 55 T**),
Test for Chloride Ion in Industrial Water and Industrial Waste Water (**D 512 - 55 T**),
Test for Silica in Industrial Water and Industrial Waste Water (**D 859 - 55 T**),
Test for Dissolved Oxygen in Industrial Water (**D 888 - 49 T**),
Test for Acidity and Basicity (Alkalinity) in Industrial Water and Industrial Waste Water (**D 1067 - 57 T**),
Test for Sodium and Potassium in Industrial Water (**D 1127 - 50 T**),
Test for Sulfite Ion in Industrial Water (**D 1339 - 54 T**),
Test for Hydrazine in Industrial Water (**D 1385 - 58 T**),
Test for Sodium and Potassium Ions in Industrial Water and Water-Formed Deposits by Flame Photometry (**D 1428 - 56 T**),
Sampling Homogeneous Industrial Waste Water (**D 1496 - 57 T**),
Test for Iron in High Purity Industrial Water (**D 1497 - 57 T**), and
Test for Effect of Water in Tubular Heat Exchangers (**D 1591 - 58 T**).

Tentative Specifications for:

Equipment for Sampling Industrial Water and Steam (**D 1192 - 51 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives and standards:

Tentative Methods of Sampling Industrial Water (D 510 - 55 T):[2]

Section 19.—Rearrange paragraphs so that reagents are listed in alphabetical sequence.

Reletter Paragraph (*b*) as (*a*) and change the heading to read: "(*a*) *Cleaning Solution*—Acid Dichromate.[5]" Add a new footnote 5 to read: "[5] Reagent used for preparation of apparatus only."

Change Paragraph (*c*) to (*b*) and revise to read as follows: "(*c*) *Dilute Nitric Acid* (*1:4*).—Prepare nitric acid (1:4) by mixing 1 volume of HNO_3 (sp gr 1.42) with 4 volumes of reagent water."

Tentative Methods of Test for Chloride Ion in Industrial Water and Industrial Waste Water (D 512 - 55 T):[2]

Section 16.—Include the reagent strength designations for Section 16 (*a*) to (*g*). Change Paragraph (*b*) to read as follows:

(*b*) *Phenolphthalein Indicator* (*5.0 g per liter*).—Dissolve 0.5 g of phenolphthalein in 50 ml of ethyl alcohol (95 per cent) (Note 7). Dilute to 100 ml with water.

NOTE 7.—Specially denatured ethyl alcohol conforming to Formula No. 3A or 30 of the U. S. Bureau of Internal Revenue may be substituted for ethyl alcohol (95 per cent).

Tentative Methods of Test for Silica in Industrial Water and Industrial Waste Water (D 859 - 55 T):[2]

Section 16.—Change Paragraph (*a*) to read as follows:

(*a*) *Amino-Naphthol Sulfonic Acid Solution.*—Dissolve 0.5 g of 1-amino-2-naphthol-4-sulfonic acid in 50 ml of a solution containing 1 g of sodium sulfite (Na_2SO_3). After dissolving, add the solution to 100 ml of a solution containing 30 g of sodium hydrogen sulfite ($NaHSO_3$). Make up to 200 ml and store in a dark plastic bottle. Prepare a fresh solution every 2 weeks.

Change Paragraph (*c*) to read as follows:

(*c*) *Buffer Solution.*—Dissolve 10 g of sodium tetraborate ($Na_2B_4O_7 \cdot 10H_2O$) in reagent water and dilute to 1 liter. Store in hard rubber or polyethylene bottle, or in a paraffin-coated chemically resistant glass bottle.

Tentative Methods of Test for Dissolved Oxygen in Industrial Water (D 888 - 49 T):[2]

Section 6.—Change Paragraph (*d*) to read as follows:

(*d*) *Manganous Sulfate Solution* (*364 g per liter*).—Dissolve 364 g of manganous sulfate ($MnSO_4 \cdot H_2O$) in water, filter, and dilute to 1 liter. No more than a trace of iodine should be liberated when the solution is added to an acidified potassium iodide (KI) solution.

Change Paragraphs (*f*), (*g*), and (*h*) to read as follows:

(*f*) *Sodium Thiosulfate Standard Solution* (*0.1 N*).—Using a 1000-ml volumetric flask, dissolve 25 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in approximately 800 ml of water that has just been boiled and cooled. Invert the flask at regular, short intervals until the solid is dissolved. Stabilize the solution by dissolving in it in the same manner, 1 g of sodium carbonate (Na_2CO_3) or 4 g of sodium tetraborate ($Na_2B_4O_7 \cdot 10H_2O$). Dilute to 1 liter with boiled water. Standardize against potassium dichromate ($K_2Cr_2O_7$) as follows:

Dissolve 0.0981 g of dried $K_2Cr_2O_7$ in water. Dissolve approximately 2 g of iodate-free KI in this mixture, then add 7 ml of HCl (sp gr 1.19) and mix. Titrate immediately with the sodium thiosulfate solution being standardized using starch as the indicator, until the color just changes from blue to the green color of the chromic salt. Calculate as follows:

$$\text{Normality of } Na_2S_2O_3 = \frac{\text{grams of } K_2Cr_2O_7 \times 1000}{(\text{equivalent of } K_2Cr_2O_7) \times S}$$

where S = milliliters of $Na_2S_2O_3$ solution required.

(*g*) *Sodium Thiosulfate, Standard Solution* (*0.005 N*).—With a calibrated pipet transfer 12.50 ml of 0.1 *N* sodium thiosulfate solution to a 250-ml volumetric flask. Dilute to the mark with water and mix completely. This solution shall be prepared not more than 12 to 15 hr before using.

(*h*) *Starch Indicator.*—Make a paste of 1 g of arrowroot starch with cold water. Pour the paste into 100 ml of boiling water and boil for several minutes. Store in a glass stoppered bottle in a cool place. Starch solution prepared in this manner will remain chemically stable for two or three days.

Standard Method of Test for Iodide and Bromide Ions in Industrial Water (D 1246 – 55):[2]

Section 6.—Change Paragraph (*j*) to read as follows: "(*j*) *Hydrochloric Acid* (*1:4*).—Mix 1 volume of HCl (sp gr 1.19) with 4 volumes of water."

Section 8(*d*).—In line 11 change "HCl (1:2)" to read "HCl (1:4)." In line 12 change "0.5 ml of HCl (1:2)" to read "1 ml of HCl (1:4)."

Tentative Method of Test for Nitrite Ion in Industrial Water (D 1254 – 57 T):[2]

Section 7.—Change Paragraph (*c*) to read as follows: "(*c*) *Sodium Acetate Solution* (*275 g per liter*).—Dissolve 27.5 g of sodium acetate trihydrate ($NaC_2H_3O_2 \cdot 3H_2O$) in water, dilute to 100 ml, and filter."

Tentative Methods of Test for Dissolved Oxygen in Industrial Waste Water (D 1589 – 58 T):[2]

Section 8.—Change Paragraph (*g*) to read as follows:

(*g*) *Starch Indicator.*—Emulsify 5 to 6 g of potato or arrowroot starch in a mortar with a few milliliters of cold water. Pour the emulsion into 1 liter of boiling water, allow to boil for a few minutes, and settle overnight. Use the clear, supernatant solution. If the starch solution is to be used for a period of several days, a preservative of 1.3 g of salicylic acid or a few drops of toluene should be added.

Section 24.—In Paragraph (*b*) change "(325 g per liter)" to read "(364 g per liter)."

Tentative Method of Test for Copper in High-Purity Water (D 1688 – 59 T):[2]

Section 7.—Change Paragraph (*j*) to read as follows:

(*j*) *Sodium Acetate Solution* (*275 g per liter*).—Dissolve 55 g of sodium acetate trihydrate ($NaC_2H_3O_2 \cdot 3H_2O$) in water and dilute to 200 ml. Remove traces of copper from the reagent by adding 1 ml of hydroxylamine hydrochloride and then proceed as described in Paragraph (*d*).

Tentative Method of Test for Silica in High-Purity Water (D 1689 - 59 T):[2]

Section 8.—At the end of Paragraphs (*b*) and (*e*), add a sentence to read: "Store in a hard rubber or polyethylene bottle or in a paraffin-coated, chemically resistant glass bottle."

At the end of Paragraph (*d*), add a sentence to read: "Store in a chemically resistant glass bottle."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

The committee accepted the recommendations concerning new tentatives, revisions of published tentatives and standards, the change of status of tentatives, published earlier in this report, from Subcommittees II, III, IV, V, VI and VII.

Subcommittee II on Definitions and General Specifications (S. K. Love, chairman).—The Task Group on Reporting Analysis was authorized to include in its program the activity of the Task Group on Reporting Analysis of Industrial Waste Water (formerly a task group of Subcommittee VII). The task group was supplied with units for reporting the results of analysis in radioactive water, as supplied by Subcommittee V.

The Task Group on Terminology and Nomenclature continued its activity on definitions supplied to the task group from other subcommittees.

The Task Group on Uniformity of Reagents was authorized to undertake the preparation of material for the inclusion of standardization procedures of the commonly used standard solutions in the methods of test of Committee D-19.

Subcommittee III on Methods of Sampling and Flow Measurement (E. F. Davidson, chairman).—The Task Group on Sampling of Steam reviewed the Standard Method of Sampling and Testing of Superheated Steam, issued by the British Standards Institution. The task group anticipates receipt of the investigation data from the U.S.N. Boiler and Turbine Laboratory, this test being made for the Steam Contamination Subcommittee of the Joint Research Committee on Boiler Feed Water Studies of the American Society of Mechanical Engineers.

The Task Group on Flow Measurement is currently studying a draft of a proposed method making use of the Parshall Flume technique.

The Task Group on Sampling for Determination of Radioactivity in Water is studying suitable methods.

Subcommittee IV on Methods of Analysis of Industrial Water (J. F. J. Thomas, chairman).—Task groups of the subcommittee are working on the following new methods and revisions of tentatives and standards, as follows: acidity and alkalinity, aluminum, ammonia, bromide and iodide, chloride in industrial water and in high-purity industrial water, copper, dissolved oxygen, fluoride, hardness, iron in industrial water and in high-purity industrial water, lead, manganese, nickel, nitrates, nitrites and organic nitrogen, phosphate, silica in industrial water and in high-purity industrial water, suspended and dissolved solids, sulfur compounds, volatile amines, and zinc.

Subcommittee V on Instrumental Analysis (A. O. Walker, chairman).—The task groups of the subcommittee are working on the following new methods and revisions of tentatives and standards: appearance properties (light transmittance and turbidity), biological microscope, electrical conductivity, flame photometric determination of sodium and potassium in industrial water and in high-purity industrial water, hydrogen, oxidation-reduction potential, polarographic testing, radioactivity measurements, surface tension, and pH.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI on Performance Testing (J. K. Rice, chairman).—The task groups of the subcommittee continue their studies for proposed methods and revisions of tentatives and standards pertaining to deposit thickness, impurities in steam, ion exchange materials performance, ion exchange materials properties, radioactivity tracer testing, service testing of tubular metallic products, and specifications for heavy water.

The Task Group on Steam Impurities is awaiting the results of the research investigation mentioned earlier in this report.

The Task Group on Specifications for Heavy Water is assembling analytical methods both within ASTM and from material published elsewhere.

Two new task groups were formed: on Radiostrontium in Industrial Water and on Reactor Cooling Water Contamination.

The Board of Directors has approved the preparation of a proposed purchase specification for heavy water.

Subcommittee VII on Methods of Analysis of Industrial Waste Water (R. A. Baker, chairman).—The task groups of the subcommittee continue their studies for new methods and revisions of tentatives and standards pertaining to acidity and basicity, chlorine, chromium, cyanides, dissolved oxygen, suspended and dissolved matter, odor, oily matter, oxygen demand (chromate oxygen demand and biological oxygen demand), phenolic compounds, specific gravity, sulfur compounds, surface active materials, and toxicity.

Subcommittee VIII on Methods of Analysis of Deposits (F. U. Neat, chairman).—The committee authorized the establishment of this subcommittee at the June 27, 1959 meeting, in Atlantic City. Exploratory work was done to ascertain the area of immediate activity of the subcommittee, and the consideration of task groups to initiate the program of the subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 144 members; 76 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

ORRIN M. ELLIOTT,
Secretary.

REPORT OF COMMITTEE D-20
ON
PLASTICS*

Committee D-20 held three meetings during the past year: during the Annual Meeting of the Society in Atlantic City, N. J., on June 21, 22, and 23, 1959; in Cleveland, Ohio on Sept. 22 to 25, 1959; and in Cincinnati, Ohio, on March 7 to 10, 1960. A special meeting of the Advisory Committee was held in Washington, D. C. on July 22, 1959.

The committee consists of 496 individuals representing 286 voting members, of whom 265 are classed as producers, 118 as consumers, and 113 as general interest members.

The work on test methods and specifications for polyolefin plastics became so extensive in 1959 that a new subcommittee (XII) was established to permit more effective prosecution of this activity, previously the responsibility of a section in another subcommittee.

Increased participation in and contribution to Committee D-20 activity by West Coast members led to the development of a policy for establishing West Coast sections, integrated into the Committee D-20 structure, to facilitate West Coast activities.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. B. De Coste.
First Vice-Chairman, P. E. Willard.
Second Vice-Chairman, J. E. Hanna.
General Secretary, C. F. Ferraro.
Membership Secretary, H. C. Gunst.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the recommendations listed below. The recommendations were accepted by the Administrative Committee on the dates indicated.

New Tentative Method of Test for:

Indentation Hardness of Plastics by Means of a Durometer (D 1706 - 59 T) (Approved Nov. 23, 1959),
Particle Size Analysis of Powdered Polymers and Copolymers of Vinyl Chloride (D 1705 - 59 T) (Approved Oct. 27, 1959),
Tensile Properties of Plastics: Microtensile Specimens (D 1708 - 59 T) (Approved Dec. 28, 1959),
Impact Resistance of Polyethylene Film by the Free Falling Dart Method (D 1709 - 59 T) (Approved Dec. 28, 1959),
Resistance of Plastics to Sulfide Staining (D 1712 - 60 T) (Approved March 23, 1960), and
Determination of Gas Evolved from Chemical Blowing Agents for Cellular Plastics (D 1715 - 60 T) (Approved March 23, 1960).

New Tentative Specifications for:

Polytetrafluoroethylene Rod (D 1710 - 60 T) (Approved March 23, 1960).

New Tentative Recommended Practice for:

Presentation of Capillary Flow Data on Molten Thermoplastics (D 1703 - 59 T) (Approved Oct. 27, 1959).

Revision of Tentative Specification for:

Polyethylene Molding and Extrusion Materials (D 1248 - 59 T) (Approved Nov. 6, 1959).

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

Revision of Tentative Method of Test for:

Water Absorption of Plastics (D 570 - 59a T) (Approved Dec. 28, 1959).

Revision and Reversion to Tentative of Standard Method of Test for:

Ammonia in Phenol-Formaldehyde Molded Materials (D 834 - 57) (Approved Nov. 6, 1959)

Haze and Luminous Transmittance of Transparent Plastics (D 1003 - 52) (Approved Oct. 27, 1959)

The new tentative methods D 1705, D 1706 and recommended practice D 1703 appear in the 1959 Supplement to Book of ASTM Standards, Part 9 together with the revised tentative methods D 570, D 834 and D 1003. The new tentative methods D 1708 and D 1709 appear in the 1959 Compilation of ASTM Standards on Plastics. The new tentative methods D 1712 and D 1715, and tentative specifications D 1710 are available as separate reprints.

AMERICAN STANDARDS

The following standards under the jurisdiction of Committee D-20 have been approved as American Standard by the American Standards Assn.:

Standard Methods of Test for:

Ammonia in Phenol-Formaldehyde Molded Materials (D 834 - 59; ASA K65.11—1960),

Deformation of Plastics Under Load (D 621 - 59; ASA K65.4—1960), and

Haze and Luminous Transmittance of Transparent Plastics (D 1003 - 59 T; ASA K65.5—1960).

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications, methods of test, and recommended practice as appended hereto:[1]

Tentative Methods of Test for:

Brittleness Temperature of Plastic Film by Impact, and

Evaluation of Welding Performance for Poly(Vinyl Chloride) Structures.

Tentative Specifications for:

Rigid Acrylonitrile-Butadiene-Styrene (ABS) Plastics,

Toluenediisocyanate and Poly(Oxypropylene Glycol),

IPS Rigid Poly(Vinyl Chloride) (PVC) Plastic Pipe, and

Rigid Poly(Vinyl Chloride) Compounds.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Tensile Properties of Plastics (D 638 - 58 T):[2]

Revise as appended hereto.[1]

Tentative Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 - 58 T):[2]

Definition of Pit.—Change to read as follows:

Pit, *n.*—Small regular or irregular crater in the surface of a plastic, usually with its width approximately of the same order of magnitude as its depth.

Tentative Specifications for Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes (D 702 - 59 T):[3]

Section 2(b).—Change Grade 3 to read as follows: "*Grade 3.*—Unshrunk sheet, having a highly polished, finished surface, with greater shrinkage than Grade 1 when heated."

Section 3.—In the first sentence of Paragraph (*b*), in the first sentence immediately following the list of sizes, and in the note following Item (*1*) change "Table I" to read "Table I and Table II."

Table I.—Change the title by the insertion of the words "Grade 1 and Grade 2" before the word "Cast."

---

[1] The new and revised tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

[2] 1958 Book of ASTM Standards, Part 9.

[3] 1959 Supplement to Book of ASTM Standards, Part 9.

New Table.—Add a new Table II to read as shown in the accompanying Table I, renumbering subsequent tables accordingly.[4]

Section 4.—In line 2 change "Table II" to read "Table III."

Section 6.—In Paragraph (*e*), the first line, change "On grade 1," to read "On grade 1 and grade 3." Change the last sentence of Paragraph (*f*) to read: "Haze shall be measured on grade 1 and grade 3 clear material only." Change the last sentence of Paragraph (*g*) to read: "Measurements shall be made on grade 1 and grade 3 sheets only."

Tentative Specifications for Melamine-Formaldehyde Molding Compounds (D 704 – 56 T):[2]

Section 2.—In the second line change "nine types" to read "seven types." Delete types 4 and 9 from the listing of types.

Table I.—Change to read as shown in the accompanying Table II.[4]

Section 6.—Change Paragraph (*d*) to read as follows: "(*d*) *Flexural Strength.*—Method of Test for Flexural Properties of Plastics (ASTM Designation: D 790)."

In Paragraph (*h*) delete the last part of the paragraph, beginning with the words "except that the test specimens . . .," and replace with the words: "using specimens measuring $\frac{1}{8}$ in. in thickness and 4 in. in diameter, tested under oil between cylindrical brass electrodes 1 in. in length and 1 in. in diameter, with edge rounded to a radius of $\frac{1}{8}$ in."

Change Paragraph (*i*) to read as follows:

> (*i*) *Loss Factor.*—Methods of Test for A-C Capacitance, Dielectric Constant, and Loss Characteristics of Electrical Insulating Materials (ASTM Designation: D 150) using a 4-in. diameter compression molded disk $\frac{1}{8}$ in. in thickness for tests at all frequencies up to and including 1 Mc.

To Paragraph (*j*) add the words "using the tungsten rod electrodes."

Tentative Definitions of Terms Relating to Plastics (D 883 – 59 T):[3]

New Definitions.—Add the following new definitions, inserting them in the proper alphabetical sequence:

Aging, *n.*—(*1*) The effect on plastics of exposure to an environment for an interval of time.

(*2*) The process of exposing plastics to an environment for an interval of time.

Artificial Weathering.—The exposure of plastics to cyclic laboratory conditions involving changes in temperature, relative humidity, and ultraviolet radiant energy, with or without direct water spray, in an attempt to produce changes in the material similar to those observed after long-term continuous outdoor exposure.

NOTE.—The laboratory exposure conditions are usually intensified beyond those encountered in actual outdoor exposure in an attempt to achieve an accelerated effect. This definition does not involve exposure to special conditions such as ozone, salt spray, industrial gases, etc.

Consistency, *n.*—The resistance of a material to flow or permanent deformation when shearing stresses are applied to it; the term is generally used with materials whose deformations are not proportional to applied stress.

NOTE.—Viscosity is generally considered to be similar internal friction that results in flow in proportion to the stress applied. (See Viscosity and Viscosity Coefficient.)

Dispersion, *n.*—A heterogeneous system in which a finely divided material is distributed in another material.

NOTE.—In plastics technology, a dispersion is usually the distribution of a finely divided solid in a liquid or a solid. For example, (1) pigments or fillers in molded plastics, (2) plastisols, or (3) organosols. A dispersion of a solid in a liquid is a suspension. (See Plastisol and Organosol.)

Isotactic, *adj.*—Pertaining to a type of polymeric molecular structure containing a sequence of regularly spaced asymmetric atoms arranged in like configuration in a polymer chain.

NOTE.—Materials containing isotactic molecules may exist in highly crystalline form because of the high degree of order that may be imparted to such structures.

---

[4] The new table is not included in this report but appears in the revised tentative; see 1960 Supplement to Book of ASTM Standards, Part 9.

Plastics Pipe.—A hollow cylinder of a plastic material in which the wall thicknesses are usually small when compared to the diameter and in which the inside and outside walls are essentially concentric.

NOTE.—Much confusion exists in industry regarding use of the terms "pipe" and "tubing." Plastics pipe is usually made from rigid or semirigid plastics while plastics tubing is usually made from nonrigid plastics, or is of small diameter.

Plastics Tubing.—See *Plastics Pipe.*

Plastisol, *n.*—A suspension of a finely divided resin in a plasticizer.

NOTE.—The resin does not dissolve appreciably in the plasticizer at room temperature but does at elevated temperatures to form a homogeneous plastic mass (plasticized resin).

Preform, *n.*—A coherent, shaped mass of powdered, granular or fibrous plastic molding compound, or of fibrous filler material with or without resin.

NOTE.—A preform may be made by compressing the material sufficiently to produce a coherent shaped mass for convenience in handling.

Revised Definitions.—Change the definitions of Postforming and Gel Point to read as follows:

Postforming, *n.*—The forming of cured thermoset plastics. (See also Forming.)

Gel Point.—The stage at which a liquid begins to exhibit pseudo-elastic properties.

NOTE.—This stage may be conveniently observed from the inflection point on a viscosity-time plot. (See Gel.)

Tentative Method of Test for Measurement of Density of Plastics by the Density-Gradient Technique (D 1505 – 57 T):[2] Revise as appended hereto.[1]

Tentative Abbreviations of Terms Relating to Plastics (D 1600 – 58 T):[2]

Section 2(a).—Add the following term and abbreviation: "Cellulose Acetate Propionate . . . . CAP."

Section 2(b).—Add the following term and abbreviation: "Triphenyl Phosphate . . . . TPP."

Section 2(c).—Add the following term and abbreviation: "Solvent Welded Plastics Pipe . . . . SWP."

Tentative Methods of Testing Urethane Foam Raw Materials (D 1638 – 59 T):[3] Revise as appended hereto.[1]

Tentative Method of Test for Environmental Stress-Cracking of Type I Ethylene Plastics (D 1693 – 59 T):[3]

Section 5.—Delete Paragraphs (*a*), (*b*), and (*c*) and footnotes included, relettering subsequent paragraphs accordingly.

Section 7.—Delete the last sentence of Paragraph (*a*).

Note 5.—Move to follow Paragraph (*a*) and change the second sentence to read: "However, inert materials such as polyester film, unplasticized cellophane, polytetrafluoroethylene, and aluminum foil have been found satisfactory."

New Note 6.—Add a new Note 6 immediately following Note 5 to read as follows, renumbering subsequent Notes accordingly:

NOTE 6.—A method found satisfactory for pressing sheet from which specimens may be cut utilizes the following apparatus and procedure:

(*a*) *Molding Chase.*—A "picture frame"-type compression molding chase having a blanked out section of suitable size[2] and a thickness to produce molded slabs 0.125 ± 0.005 in. (Dimensions shown in Fig. 1 are for mold to produce 6 by 6 in. sheets.)

(*b*) *Mold Release Agent.*—Clean aluminum foil[3] at least 0.003 in. thick, and of area adequate to cover the molding chase of (*a*).

(*c*) *Molding Plates.*—Two polished chromium-plated ferrotype plates (Fig. 1), such as used in photography, at least 0.040 in. thick, and of area adequate to cover the molding chase of (*a*).

Clean with solvent and dry a polished ferrotype molding plate. Lay down and smoothly cover the plate with a sheet of aluminum foil mold release agent also cleaned and dried. Place the mold chase on top of this assembly. Place within the mold chase a suitable quantity of the plastic pellets, granules, or milled crepe to completely fill the blanked out area when molded. A slight excess is desirable.[4] Smooth the granules so as to form a uniform layer. Place a second sheet of cleaned and dried aluminum foil on top of the granules and add the clean dry mold plate. Place the whole assembly in a hot compression molding press and mold to a sheet of the specified size. This is a flash-type molding and excess material should flow out between the chase and platens. The molding temperature, as measured by pyrometer on the mold platens, should be 165 C. It is desirable to hold the loaded press closed for about 2 min at 165 C prior to applying pressure. Use suitable pressure to form a smooth, void-free sheet. Introduce cooling water at rates such that the sheet is cooled from 165 C to 75 C at a reasonably uniform rate of 6 to 11 C per min. The sheet should be removed from the press at 75 C or lower. Do not cut specimens from any area within 1 in. of the edge of the sheet.

---

[2] A 6 by 6 in. blanked out section has been found satisfactory.

[3] Alloy 1145 of temper H19, as supplied by the Aluminum Co. of America, has been found satisfactory.

[4] It has been found that 75 ± 1 g is a satisfactory charge for a 6 by 6 in. sheet.

Delete Paragraph (*b*) and footnote, renumbering Paragraph (*c*) as (*b*).

Section 11.—Add a new Item (*2*) to read as follows, renumbering subsequent items accordingly: "(*2*) Manner of preparation of test sheets (i.e., from granules or from milled crepe)."

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revision as indicated in the Report of Committee D-9 on Electrical Insulating Materials of the Standard Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (**D 785 – 51**)[2] and reversion of the method to tentative. This standard is under the joint jurisdiction of ASTM Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Dilute Solution Viscosity of Vinyl Chloride Polymers (**D 1243 – 58 T**),[2]
Diffuse Light Transmission Factor of Reinforced Plastic Panels (**D 1494 – 57 T**),[2]
Transverse Load of Corrugated Reinforced Plastic Panels (**D 1502 – 57 T**),[2] and
Bearing Load of Corrugated Plastic Panels (**D 1602 – 58 T**).[2]

Tentative Specification for:

Cellulose Acetate Sheet and Film for Primary Insulation (**D 1202 – 52 T**),[2] subject to action by Committee D-9 on Electrical Insulating Materials,
Styrene-Acrylonitrile Copolymer Molding and Extrusion Materials (**D 1431 – 56 T**),[2] and
Cellulose Propionate Molding and Extrusion Materials (**D 1562 – 58 T**).[2]

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Test for Diffusion of Light by Plastics (**D 636 – 54**).[2]

WITHDRAWAL OF TENTATIVES

The committee recommends the withdrawal of the following tentatives:

Tentative Specifications for:

Methylstyrene-Acrylonitrile Copolymer Molding and Extrusion Materials (**D 1594 – 58 T**),[2] and
Polymethylstyrene Molding and Extrusion Material (**D 1595 – 58 T**).[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Resistance of Plastics to Chemical Reagents (**D 543 – 56 T**),
Colorfastness of Plastics to Light (**D 620 – 57 T**),

Flammability of Rigid Plastics over 0.050 in. in Thickness (**D 635 - 56 T**),
Repeated Flexural Stress (Fatigue) of Plastics (**D 671 - 51 T**),
Brittleness Temperature of Plastics and Elastomers by Impact (**D 746 - 57 T**),
Stiffness in Flexure of Plastics (**D 747 - 58 T**),
Tensile Properties of Thin Plastic Sheets and Films (**D 882 - 56 T**),
Measuring Flow Rates of Thermoplastics by Extrusion Plastomer (**D 1238 - 57 T**),
Orientation Release Stress of Plastic Sheeting (**D 1504 - 57 T**),
Vicat Softening Point of Plastics (**D 1525 - 58 T**),
Determination of Bierbaum Scratch Hardness of Plastic Materials (**D 1526 - 58 T**),
Tensile Modulus of Elasticity of Thin Plastic Sheeting (**D 1530 - 58 T**), and
Determining Carbon Black in Ethylene Plastics (**D 1603 - 58 T**).

Tentative Specifications for:

Phenolic Molding Compounds (**D 700 - 57 T**),
Polystyrene Molding and Extrusion Materials (**D 703 - 56 T**),
Urea-Formaldehyde Molding Compounds (**D 705 - 58 T**),
Cellulose Acetate Molding and Extrusion Compounds (**D 706 - 55 T**),
Vinyl Chloride Polymer and Copolymer Rigid Sheets (**D 708 - 53 T**),
Laminated Thermosetting Materials (**D 709 - 55 T**),
Methacrylate Molding and Extrusion Compounds (**D 788 - 56 T**),
Tetrafluoroethylene Resin Molding and Extrusion Materials (**D 1457 - 56 T**), and
Glass Fabric Reinforced Epoxy Resin Laminates (**D 1592 - 58 T**).

Tentative Recommended Practice for:

Accelerated Weathering of Plastics Using S-1 Bulb and Fog Chamber (**D 795 - 57 T**),
Injection Molding of Specimens of Thermoplastic Materials (**D 1130 - 50 T**), and
Resistance of Plastics to Artificial Weathering Using Fluorescent Sun Lamp and Fog Chamber (**D 1501 - 57 T**).

Tentative Specifications and Methods of Test for:

Laminated Thermosetting Decorative Sheets (**D 1300 - 53 T**),
Polychlorotrifluoroethylene Molding and Extrusion Materials (**D 1430 - 58 T**), and
Random Chopped Glass Fiber Reinforcing Mat (**D 1529 - 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties (P. Z. Adelstein, chairman).—Revision of the tensile definitions appended to Method D 638 has been completed. Two new tentatives that have been approved are a test for the indentation hardness of plastics (Shore A and D) (D 1706) and a tension test using micro specimens (D 1708). Work completed includes a revision to the Test for Rockwell Hardness of Plastics (D 785) and a change in the tolerances of the specimens in the test for Tensile Properties of Plastics (D 638). A successful testing program has been completed on a tension impact test and the working draft is being balloted. Round-robin test programs have also been completed on the measurement of blocking resistance of polyethylene, the friction of plastic films, and the Elmendorf Tear Test (D 689). As a result of this work, the working drafts of these three methods are being revised prior to submission for balloting. The test methods for Stiffness of Nonrigid Plastics by Means of a Torsional Test (D 1043), Stiffness in Flexure (D 747), Tensile Properties of Plastic Films (D 882), and Compressive Properties of Rigid Plastics (D 695) are currently under active study since they have become outdated and major changes are needed.

Subcommittee II on Radiation Methods (D. J. Metz, chairman), a joint subcommittee with Committee D-9, has circulated in a subcommittee letter ballot a revised version of the Tentative Recommended Practice for Exposure of Polymeric Materials to High Energy

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Radiation (D 1672). This revision was undertaken by the Section on Exposure Methods in order to bring some of the statements and concepts in the recommended practice up to date with experience gained after the original writing.

A Symposium on Postirradiation Effects in Polymers was held during the Third Pacific Area National Meeting at San Francisco in October, 1959.[6] The program consisted of an introductory paper outlining the significance of postirradiation processes to ASTM test methods, one paper on the detection of trapped free radicals by electron spin resonance measurements, and three papers dealing with measured changes in physical and chemical properties in irradiated polymers as a function of time after irradiation.

During the past year an active liaison with Committee D-11 on Rubber and Rubber-Like Materials has been established, at the request of the latter group. The subcommittee presently has 10 to 15 members of Committee D-11 on its mailing list, and looks forward to cooperation with this committee.

Subcommittee III on Thermal Properties (A. A. Harban, chairman).—A new flammability test for plastics foams and sheeting (D 1692 – 59 T) has been found to be a convenient laboratory tool for comparing the relative flammability of such materials and as the technique also appears useful for rigid plastics, a task group is currently considering the revision of other flammability Methods D 635 and D 568 along similar lines. Because of the greatly increasing interest in the rheological properties of molten plastics, a Tentative Recommended Practice for Presentation of Capillary Flow Data on Molten Thermoplastics (D 1703 – 59 T) was prepared, and was accepted by the Standards Committee. The purpose of this tentative is to promote uniform methods of reporting data and to outline possible sources of experimental error. A tentative procedure for measuring brittleness temperature of plastics films (based on the SPI test) has been submitted to Committee D-20. Other methods currently being revised include Methods D 1238 on Flow of Thermoplastics, and D 746 on Brittleness Temperature.

The Section on Ablation Tests established last year has been studying various methods using plasmajets and high temperature flames for laboratory evaluation of ablation resistance of plastics. The subcommittee was responsible for a two-session symposium on Ablation Testing[7] at the Third Pacific Area National Meeting in October 1959.

Subcommittee IV on Optical Properties (D. A. Popielski, chairman).—An objective test method, based on specular transmittance, has been developed for describing the optical clarity property (see-through distinctiveness of detail) of plastic films. This proposed method of test for transparency of plastic sheeting has been recommended for committee letter ballot.

Work on the development of an instrumental method for the measurement of yellowness of transparent and opaque plastic materials is essentially complete.

The revision of Method D 1003 accepted by the Standards Committee incorporates revisions dealing with the use of the recording spectrophotometer and accounts for designation of the preferred light source.

Subcommittee V on Permanence Properties (B. G. Achhammer, chairman) is working on a proposed tentative method of test for determining degree of exposure under artificial weathering conditions using dyed acrylic plastic for comparing relative rates of exposure. It is proposed that the dyed acrylic can be used in water-exposure apparatus and thus have an advantage over standard fading paper. Two methods for determining corrosivity of plastics, (1) galvanic action of dissimilar metals, and (2) conductivity of water extract, are being developed to provide standard methods for reporting corrosivity. A round robin on the staining of vinyl tile by rubber is in progress. A proposed tentative method of test for resistance of plastics to microorganisms is being prepared.

---

[6] The papers presented at this symposium will be included in *ASTM STP No. 276.*

[7] The papers presented at this symposium will be published as *ASTM STP No. 279.*

Method D 543 is being revised to indicate various standard specimens and test methods for measuring the effects of chemical reagents on mechanical properties and to revise and augment the list of reagents recommended for conducting resistance to chemical reagent tests.

A revision of Method D 1434 will be initiated to permit converting gas transmission rate to a unit thickness basis, to evaluate the method for cell calibration, and to resolve questions which have arisen on this method.

Method D 620 is being reviewed to decide if use of the S-1 sunlamp in this method should be continued. A new Section V-N, Environmental Stress Cracking, was established with the objective of developing test methods for determining the effect of chemicals and environments on the stress cracking of plastics.

Subcommittee VII on Analytical Methods (J. B. De Coste, chairman) is developing physical and spectrographic test methods. Section A, Physical Methods, has a revision of the specific gravity Method D 792 and a compilation of bulk density Methods D 392, D 954, and D 1182 ready for committee letter ballot. This section also had a revision of Method D 1505 on the density gradient technique accepted by the Society.

The efforts of Section H have been extended to the field of infrared analysis, with task groups studying the standardization of methods for polyolefins and poly(vinyl chloride) plastics. Information obtained from the various round robins on the optical measurement of the degree of carbon black dispersion in ethylene polymers is being reviewed for possible publication.

A revision of Method D 834 prepared by Section K, Chemical Methods, to include an alternate procedure for the determination of ammonia in phenolic plastics passed D-20 letter ballot and was accepted by the Society. Nitrogen purity and alternate means of obtaining it are the subject of a revision of Method D 1603 for the determination of carbon in ethylene polymers.

Subcommittee VIII on Research (A. G. H. Dietz, chairman) held two sessions during the year. At the first one, on the evening of Sept. 23, 1959, the following four papers were presented: "A New Stress-Cracking Test for Polyolefins" by F. J. McGarry and W. J. Sauber; "The Philosophy of the Evaluation of Fracture Resistance of Materials" by Joseph A. Kies; "Flexural and Impact Variations of Phenolic Moldings: A Statistical Round-Robin Study" by Curtis Elmer and E. C. Harrington; and "A Vicat-Type Penetration Tester for Evaluating Hardness and Elastic Recovery in Polymeric Materials" by F. W. Adams. At the Spring meeting, March 8, Mr. Neil Connor of the Federal Housing Administration discussed the problems of the FHA in establishing standards for plastics products to be used in house construction. Mr. Frank Reinhart also entered into the discussion from the standpoint of developing the Commercial Standards as well as ASTM Standards.

Subcommittee IX on Molds and Molding (G. H. Williams, Jr., chairman).—A revision of Recommended Practice D 796 is in process. A new recommended practice for transfer molding test specimens of phenolic materials is being prepared. The evaluation of a proposed standard procedure for the injection molding of test specimens on miscellaneous commercial machines continues. A second round robin, more than half completed, still shows encouraging results. The potentialities for standardization of molding procedures on "miniature" injection molding machines is progressing. Difficulties reported on the ejection of the microtensile specimens have been overcome. The round robin originally proposed for the evaluation of the microtensile specimens prepared on small machines will be carried out.

The development of mold release testing procedures is being discussed and a mold has been designed to obtain data on the several test methods suggested.

A new section on extrusion processes has been formed and is currently defining its proposed scope of activity.

Subcommittee X on Definitions (P. E. Willard, chairman) is continuing work on the development of definitions of terms relating to plastics and of abbreviations for plastics terms. Liaison has been established with Committee D-11 on Rubber and Rubber-Like Materials in an effort to eliminate conflict on definitions used in the rubber and plastics fields. A proposal for the combination of all definitions for plastics terms into a single standard comprising a "plastics dictionary" has been made and work is proceeding on such a compilation.

Subcommittee XII on Polyolefin Plastics (J. B. Howard, chairman) was created by the D-20 Advisory Committee at the 1959 Annual Meeting by expanding the scope of what had been Section J of Subcommittee XV to include all polyolefin plastics. Its initial contributions led to the acceptance of Method D 1693 known as the bent strip test for environmental stress cracking of ethylene plastics and revisions of the method for solution viscosity of ethylene plastics as well as of the polyethylene materials specification, which have become, respectively, Method D 1601 and Specifications D 1248.

Currently under study are two alternative stress crack procedures; methods for determining oxidative stability, for tensile testing, and for preparation and conditioning of test specimens; addition of a stress crack resistant grade to Specifications D 1248; and preparation of a polypropylene materials specification with associated test procedures.

The subcommittee's most notable accomplishment to date is probably the acceptance as a tentative of the bent-strip environmental stress crack test. This procedure has been in successful use by the industry for over 10 years in the qualitative form finally adopted by committee D-20 after more than 5 years of unsuccessful endeavor to make it more quantitative. It has only recently become evident that inability to reproduce test sheets of polyethylene universally on a precise basis has probably been the major obstacle, and that this inability can markedly affect all tests for crystallinity sensitive properties of polyethylene. There appears to be justification for optimism that the studies of conditioning procedures now under way in Subcommittee XII may lead to substantial improvements in this regard.

Subcommittee XIII on Statistical Techniques (R. H. Supnik, chairman) has made excellent progress in writing recommended practices on (1) sampling and (2) experimental design. Several drafts have been written and final modifications are now being made.

A survey was made of all plastics test methods under Committee D-20 jurisdiction. The objective was to summarize the manner in which these methods were written with respect to the following items: (1) number of test specimens specified, (2) sampling procedure, (3) instructions for discarding and re-test, (4) values to be reported, and (5) discussion of precision and reproducibility. It was found that there was no uniformity in statistical treatment and in many instances the manner of handling the above items was grossly inadequate.

The series of indoctrination lectures, initiated last year, met with tremendous reception this year. These lectures are designed to provide Committee D-20 members with an understanding of statistical considerations and concepts as they may be applied to writing test methods and specifications. The speakers and their subjects were as follows: "Sampling Plans," by Spencer M. Free, Jr., SKF Laboratories, Philadelphia (June, Atlantic City); "Non-parametric Techniques," by Arthur Zoellner, General Electric Co., Schenectady (September, Cleveland); and "Statistical Approach to Materials Specifications," by William R. Pabst, Jr., U. S. Navy Department, Bureau of Ordnance (March, Cincinnati).

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman) has in preparation a tentative revision of Methods D 618 in which additional standard test temperatures, higher than those now recommended, are proposed for conditioning or testing higher temperature-resistant plastics and insulating materials. The work of this subcommittee is a joint activity of Committees D-9 and D-20.

Subcommittee XV on Thermoplastic Materials (L. Gilman, chairman) recommended that Specifications D 1431 and D 1562 be advanced to standard; that Specifications D 703, D 706, D 788, D 1430, and D 1457 be continued as tentatives because they are currently under revision; that Specifications D 1594 and D 1595 be discontinued because no longer manufactured; and that proposed specifications for polytetrafluoroethylene rod, rigid vinyl plastic compounds, and rigid acrylonitrile-butadiene-styrene plastics be accepted as tentatives, as noted earlier in the report. The latter three are important commercial materials for which specifications were requested some time ago and are needed.

Subcommittee XVI on Thermosetting Materials (J. E. Hanna, chairman) has prepared a new tentative specification for epoxy casting resins and a revision of Specification D 704.

A new Section F on Urethane Materials, under the chairmanship of S. Steingiser, has been organized and started work.

The subcommittee is engaged in a complete review of all its specifications with the objective of making them more consistent.

Subcommittee XVII on Thermoplastic Pipe and Fittings (G. H. Reed, chairman) is a joint activity of the Society of the Plastics Industry and ASTM Committee D-20. It includes nine sections, and it is anticipated that later in the year a tenth section on Drain and Conduit Piping will be organized.

The joint subcommittee of 140 members has established tentative ASTM standards on ABS plastic pipe (IPS dimensions), ABS pipe (SWP dimensions), and cellulose acetate butyrate pipe (SWP dimensions), time-to-failure of plastic pipe under long-term hydrostatic pressure and proposed tentative method of test on time-to-failure of plastic pipe under short-term hydrostatic pressure. The subcommittee prepared the Tentative Specification on Rigid PVC Pipe (IPS Dimensions) noted above and a specification for schedule A thin wall PVC pipe (IPS OD's) is ready for Committee D-20 letter ballot.

Specifications on low-density, medium-density, and high-density polyethylene pipe are being prepared as well as specifications on threaded and socket weld ABS and PVC fittings, and also specifications on insert type fittings.

A round-robin program on chemical resistance of plastic pipe and fittings is about to get under way. The subcommittee voted to request Committee D-20 to request active participation through the American Standards Assn. in ISO/TC 5/SC 6 on Plastic Pipe.

Subcommittee XVIII on Reinforced Plastics (H. A. Perry, chairman) pursued methods and specifications activities during the year on flat sheets, pipe and fittings, corrugated panels, laminating resins, reinforcements, and high-temperature resistant materials. Work was undertaken toward the improvement and revision of Specifications D 709, D 1300, and D 1592. Work was begun on a proposed tentative specification for corrugated panels. The proposed tentative specification for woven glass fabric, cleaned and after-finished, for plastic laminates had been revised for joint subcommittee and committee ballot. A proposed tentative specification for woven roving glass fabric for plastic laminates has been sent out to subcommittee ballot. A working group was formed to develop methods of test for "wet-out" and "resin-pickup" properties of reinforcements.

Section G on High-Temperature Resistant Materials has been reorganized in a dual system, Section GE (Eastern) and Section GW (Western), to expedite the handling of committee work on a nationwide basis. Five specifications and methods of test, which had been originated by Section GW, were sent to subcommittee letter ballot: (1) General Test Procedures for Determining Mechanical Properties of Plastics at Subnormal and Supernormal Temperature (−270 to +550 C); (2) Proposed Specification for Enclosures and Servicing Units for Tests at Temperatures of −270 to +550 C; (3) Recommended Practice for Determining the Effect of Heat on Plastics; (4) Proposed Tentative Method of Test for Interlaminar Shear and Secondary Bond Shear Strength of Plastic Laminates at Temperatures from −270 to +550 C; and (5) Proposed Tentative Method of Test for Shear (Johnson Double Shear) Strength of Plastics at Temperatures from −270 to +550 C.

The subcommittee arranged a Symposium on Reinforced Plastics[7] which was held at the Third Pacific Area National Meeting in October, 1959 in San Francisco. The subcommittee also cooperated with Subcommittee III in arranging a Symposium on Ablation Testing[7] at the same meeting.

Subcommittee XVIII undertook a preliminary study of new areas of work and has requested permission to undertake work in the fields of sample cutting from large moldings, procedures for the preparation of laboratory test panels, inspection procedures including classifications of defects, nondestructive test methods, methods of test and specifications for preimpregnated reinforcements and premixed roving and fabric reinforced molding compounds.

Subcommittee XIX on Plastic Film and Sheeting (K. A. Kaufmann, chairman).—Work is continuing in Section A on a specification for rigid poly(vinyl chloride) plastic sheet, and a test method for determining welding performance of poly(vinyl chloride) structures is being recommended for publication as tentative, as noted above. Work on a specification for rigid vinyl copolymer sheet is being delayed by the lack of a suitable material specification. Section B prepared the now approved Tentative Method of Test for Impact Resistance of

Polyethylene Film by the Free Falling Dart Method (D 1709) and is nearing completion of work on the coefficient of friction of polyethylene film. Work continues on development of a polyethylene film specification as satisfactory test methods are adopted. Further minor revision of Specification D 702 is in progress in Section D. Section F is actively developing a test method for determining the sealability of polyethylene film. Section E on polystyrene film and sheeting was inactivated during the year.

Subcommittee XX on Cellular Plastics (S. Steingiser, chairman) developed the tentative procedure for the determination of gas evolved from chemical blowing agents accepted by the Standards Committee and tentative specifications for toluenediisocyanate and poly(oxypropylene glycol) appended to this report.

Tentative specifications for cellular polystyrene and two new methods, one on acidity of toluenediisocyanates and the other on sodium and potassium content of polyols, to be added to Methods D 1638 are being balloted by the committee.

The subcommittee is developing methods for water absorption, water vapor transmission, open and closed cells, thermal conductivity, volume by displacement, flexure, and dimensional stability. The subcommittee is cooperating with Committee C-16 on tests for thermal insulating properties.

Methods on expandable polystyrene beads are being initiated, and a new specification on rigid urethane material is being developed as rapidly as realistic data can be assembled.

Subcommittee XXI, U.S.A. National Committee, ISO/TC 61 (W. E. Brown, chairman).—Delegates from countries representing more than half the world's population met in Munich, Germany, October 26–31 at sessions of ISO/TC 61, the International Organization for Standardization Technical Committee on Plastics. Continuing the work of developing commercially useful standards for plastics, more delegates from more nations worked on more subjects than ever before. Most significant was TC 61's continued production of Draft ISO Recommendations for test methods and nomenclature. Several of these internationally agreed upon methods are in print and several more will be available soon.

Typical was the progress made by the new working group on specifications in its first meeting since it was organized a year ago in Washington, D. C. Representatives of 12 nations agreed on how plastics specifications should be written and what materials should first be considered.

Eight other working groups approved 14 methods to be sent to the committee for balloting. In addition, 37 items on the program of work were discussed.

Four new Draft ISO Recommendations were approved at Munich for letter ballot of the 41 ISO member countries: (1) determination of the Vicat softening point, (2) viscosity number of polyamide resins in solution, (3) determination of the acetone soluble matter in phenolic molding materials, and (4) thermal stability of poly(vinyl chloride) and related copolymers and their compounds by the discoloration method. These bring to 33 the total of Draft ISO Recommendations approved by TC 61 for final preparation as internationally sanctioned methods.

Six Draft Proposals (forerunners of Draft ISO Recommendations) were approved for letter ballot of the 34 member nations: (1) method of test for tensile properties of plastics, (2) maximum temperature and time taken for temperature to rise during setting of unsaturated polyester resins, (3) determination of stiffness properties of plastics as a function of temperature by means of a torsion test, (4) resistance of plastics to natural light, (5) changes in mechanical properties after contact with chemical substances, and (6) resistance of plastics to light from the carbon arc.

Subcommittee XXII on Plastics for Tooling (J. J. Mele, chairman).—All of the five sections of Subcommittee XXII have been activated. Each of the section chairmen will select an assistant to act as co-chairman and secretary. They will also review the scopes of their respective sections. Whenever possible each section will hold special meetings in addition to regular Committee D-20 meetings. This action was thought necessary to expedite the activity of the subcommittee.

Several SPI casting test methods will be prepared for subcommittee ballot. Included in the ballot distribution will be all material formulators and consumers whose expressions of opinions the committee feels will be helpful.

This report has been submitted to letter ballot of the committee, which consists of 286 voting members; 216 members returned their ballots, of whom 203 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

F. W. REINHART,
Chairman.

R. M. BERG,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:
Poly (Vinyl Chloride) Resins (D 1755 – 60 T), and
Epoxy Resins (D 1763 – 60 T).

New Tentative Method of Test for:
Transparency of Plastic Sheeting (D 1746 – 60 T).

Revision of Tentative Specification for:
Polyethylene Molding and Extrusion Materials (D 1248 – 59 T).

Revision of Tentative Method of Test for:
Resistance of Plastics to Chemical Reagents (D 543 – 56 T), and
Carbon Black in Ethylene Plastics (D 1603 – 58 T).

Revision of Tentative Definitions of Terms Relating to:
Plastics (D 883 – 60 T).

Revision and Reversion to Tentative of Standard Methods of Test for:
Specific Gravity of Plastics (D 792 – 50).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the new and revised tentative specifications and methods appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

REPORT OF COMMITTEE D-21

ON

WAX POLISHES AND RELATED MATERIALS*

Committee D-21 on Wax Polishes and Related Materials held one meeting during the year: in Washington, D. C., on Dec. 10, 1959. In addition, the subcommittees met in Chicago, Ill., on May 19, 1960.

Additions and changes during the year brought the membership of the committee to 70, of whom 32 are classified as producers, 12 as consumers, and 26 as general interest members, with one consulting member.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. W. Walton.
Vice-Chairman, J. V. Steinle.
Secretary, B. S. Johnson.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of Test for:

Accelerated Aging of Liquid Water-Emulsion Floor Polishes,
Water Spotting of Emulsion Floor Polishes, and
Potential Long-Term Removability Properties of Water-Emulsion Floor Polishes.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Nonvolatile Matter (Total Solids) in Water-Emulsion Waxes (**D 1289 - 55 T**),[2]
60-deg Specular Gloss of Emulsion Floor Polish (**D 1455 - 56 T**),[2] and
Flash Point of Solvent-Type Liquid Waxes (**D 1437 - 56 T**).[2]

Tentative Method for:

Application of Emulsion Floor Polishes to Substrates for Testing Purposes (**D 1436 - 56 T**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (J. V. Steinle, chairman) has received approval on ten definitions for inclusion in a glossary of terms relating to wax polishes. Definitions for several additional terms are now being studied.

Subcommittee II on Raw Materials (Herbert Mellan, chairman) is working on several test methods for various properties of natural waxes, including a method for the determination of resin content of carnauba wax.

Subcommittee III on Physical and Chemical Testing (J. V. Kelly, chairman) has completed the Method of Test for Accelerated Aging of Liquid Water-

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Book of ASTM Standards, Part 10.

[2] 1958 Book of ASTM Standards, Part 10.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Emulsion Floor Polishes which is referred to earlier in this report.

Task forces are working on several other new methods, including one for the determination of silicones in wax polishes.

Subcommittee IV on Performance Tests (C. S. Kimball, chairman) has completed test methods for the determination of water spotting and removability properties of water emulsion polishes. These methods are referred to earlier in this report.

Task groups are investigating methods for the determination of discoloration and the powdering properties of emulsion waxes. In addition, the Task Group on Slip Resistance is endeavoring to develop a secondary standard for use in the calibration of the James and Sigler machines.

Subcommittee V on Specifications (William Joy, chairman) is preparing a specification for industrial type water emulsion floor waxes. A task group is also working on the preliminary draft of a specification for carnauba wax.

This report has been submitted to letter ballot of the committee, which consists of 70 members; 47 members returned their ballots, of whom 41 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. W. WALTON,
Chairman.

BAYARD S. JOHNSON,
Secretary.

REPORT OF COMMITTEE D-22

ON

METHODS OF ATMOSPHERIC SAMPLING AND ANALYSIS*

Committee D-22 on Methods of Atmospheric Sampling and Analysis held four meetings since the last report of the committee: on June 23 and 24, 1958, in conjunction with the 61st Annual Meeting of the Society in Boston, Mass.; on April 6, 1959, in Boston, Mass.; on Sept. 14, 1959, in Philadelphia, Pa.; and on April 28, 1960, in Rochester, N. Y.

The committee's membership at the time of the April 1960 meeting totaled 66.

The committee sponsored a Symposium on Air Pollution Control at the Third Pacific Area National Meeting on Oct. 16, 1959. Proceedings of the Symposium are being published as *STP No. 281.*

The committee was represented by M. D. Thomas and Morris Katz at a meeting called by the American Public Health Assn. in New Orleans on March 21, 1960. The purpose of the meeting, at which all the major national interests in air pollution problems were represented, was to discuss means for accelerating the establishment of national standards for analysis of the atmosphere. While there was general agreement that these efforts should be accelerated, it was brought out that Committee D-22 was established for this very purpose and endeavors to represent all the national interests among its membership. The committee is surveying its membership to determine whether all the interests are properly represented and is suggesting that member organizations appoint representatives who can participate actively.

The officers elected for the ensuing term of two years are as follows:

Chairman, Jacob Cholak.
Vice-Chairman, M. D. Thomas.
Secretary, August T. Rossano, Jr.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1958 Annual Meeting, the committee presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Particulate Matter in the Atmosphere (Optical Density of Filtered Deposit). This recommendation was accepted by the Standards Committee on October 2, 1959, and the new tentative is available as a separate reprint bearing the designation D 1704 – 59 T.

New Tentative

The committee recommends for publication as tentative the proposed Method for Collection and Analysis of Dustfall as appended hereto.[1]

Adoption of Tentatives as Standards Without Revision

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 10.

Tentative Method of Test for:

Concentration of Odorous Vapors (Adsorption Method) (**D 1354 - 55 T**),

Inorganic Fluoride in the Atmosphere (**D 1606 - 58 T**),

Nitrogen Dioxide and Nitric Oxide Content of the Atmosphere (Modified Griess-Ilosvay Reaction) (**D 1607 - 58 T**),

Oxides of Nitrogen in Gaseous Combustion Products (Phenol-Disulfonic Acid Procedure) (**D 1608 - 58 T**), and

Oxidant (Ozone) Content of the Atmosphere (**D 1609 - 58 T**).

Tentative Methods for:

Continuous Analysis and Automatic Recording of the Sulfur Dioxide Content of the Atmosphere (**D 1355 - 58 T**).

Tentative Definition of Terms Relating to:

Atmospheric Sampling and Analysis (**D 1356 - 58 T**).

Tentative Recommended Practices for:

Sampling Atmospheres for Analysis of Gases and Vapors (**D 1605 - 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Units (E. R. Hendrickson, chairman) is continuing the study of a list of conversion units and factors which, when established as a standard, will provide a common basis for calculations and expression of results of atmospheric analysis.

Subcommittee II on Sampling and Analysis (J. Cholak, chairman) developed the proposed Tentative Method for Collection and Analysis of Dustfall referred to above.

Task groups are developing chemical methods for determining sulfur dioxide, aldehydes, and procedures for particulate matter sampling and stack sampling.

The Task Group on Stack Sampling (H. M. Chapman, chairman) is evaluating several promising procedures for stack sampling in order to establish one which can be recommended as tentative.

In its future work, the subcommittee will give priority to development of methods for sulfur dioxide, aldehydes, carbon monoxide, carbon dioxide, and hydrocarbons among the gases. High priority was also given to the following as particulate matter: sulfuric acid, lead, and polycyclic hydrocarbons.

The Task Group on Sampling for Particulate Matter (W. J. Smith, chairman) has drafted a procedure involving special methods such as filtration, impaction, and thermal precipitation.

Mr. Harlow Chapman has been appointed chairman of Subcommittee II, taking the place of Prof. Cholak, who was elected chairman of the committee.

Subcommittee III on Instrumentation (M. D. Thomas, chairman) completed the Method of Test for Particulate Matter in the Atmosphere (Optical Density of Filtered Deposit) (D 1704 - 59 T) accepted in 1959 by the Standards Committee. Five projects are currently under way:

1. Development of a method for mass concentration of particulate matter in the atmosphere, using light scattering.

2. Instrumentation for particle size distribution measurements, also by light scattering.

3. Instrumentation for dustfall analysis according to wind direction and velocity.

4. Instrumentation for continuous measurement of nitric oxide and nitrogen dioxide in the atmosphere. This instrument method will supplement the chemical methods for nitrogen oxide D 1607 and D 1608.

5. Instrumentation for continuous

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

analysis and automatic recording of the oxidant content of the atmosphere. This also will supplement the chemical methods for oxidants (D 1609).

This report has been submitted to letter ballot of the committee, which consists of 66 voting members; 33 members returned their ballots, of whom 30 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

LESLIE SILVERMAN,
Chairman.

A. T. ROSSANO, JR.,
Secretary.

REPORT OF COMMITTEE D-23
ON
CELLULOSE AND CELLULOSE DERIVATIVES*

Committee D-23 on Cellulose and Cellulose Derivatives held one meeting during the year: in Atlantic City, N. J., on Sept. 16, 1959. The Executive Subcommittee also met in New York City on Feb. 24, 1959.

The committee consists of 45 members, of whom 13 are classified as producers, 11 as consumers, and 21 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, F. A. Simmonds.
Vice-Chairman, Kyle Ward, Jr.
Secretary, W. W. Becker.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods of test as appended hereto:[1]

Tentative Method of Test for:

Intrinsic Viscosity of Cellulose,
Cellulose Chain Length Uniformity by Fractional Precipitation of Cellulose Nitrates, and
Alcohol-Benzene Soluble Matter in Cellulose.

REVISION OF TENTATIVES

The committee recommends revisions as follows of the Tentative Definitions of Terms Relating to Cellulose and Cellulose Derivatives (**D 1695 - 59 T**)[2] and continuation of the Definitions as tentative:

(*A*) *Cellulosic Materials and Constituents.*—Change the definition of Alpha-Cellulose to read as follows:

Alpha-Cellulose.—That portion of industrial cellulosic pulps that remains insoluble after treatment with aqueous sodium hydroxide at 20 C. The term has meaning only if there is a precise statement of the initial sodium hydroxide concentration used (usually about 18 per cent), the subsequent dilution concentrations, and other conditions employed in its isolation or determination.

Delete the definition for Noncellulosic Polyose.

(*B*) *Chemical Modifications and Derivatives of Cellulose.*—Delete the definitions for Cellulose Gum and Sodium Cellulose Glycolate.

Revise the following terms as indicated:

O-Ethyl Cellulose—Change to: Ethylcellulose.
O-(2-Hydroxyethyl) Cellulose—Change to: (2-Hydroxyethyl)cellulose.
O-(2-Hydroxyethyl)-*O*-Ethylcellulose — Change to: (2-Hydroxyethyl)ethylcellulose.
O - (2 - Hydroxyethyl) - *O* - Methylcellulose—Change to: (2-Hydroxyethyl)methylcellulose.
O - (3 - Hydroxypropyl) - *O* - Methylcellulose—Change to: (2-Hydroxypropyl)methylcellulose.
O-Methylcellulose—Change to: Methylcellulose.
O-Methyl-*O*-Ethyl Cellulose—Change to: Methylethylcellulose.

Insert alphabetically a new definition to read as follows:

Propylene Glycol Ether of Methylcellulose.—Same as (2-Hydroxypropyl)methylcellulose.

(*C*) *Properties of Cellulose and Associated Concepts.*—To the definition of

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

[2] 1959 Supplement to Book of ASTM Standards, Part 6.

Haze add the following sentence to clarify the definition: "This turbidity may refer to solutions or to the solid plastic."

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Testing Sodium Carboxymethylcellulose (**D 1439 - 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (Kyle Ward, Jr., chairman).—Since the publication of Tentative Definitions of Terms Relating to Cellulose and Cellulose Derivatives (D 1695 - 59 T) several suggestions for revisions or deletions were received, as reported earlier in this report.

Subcommittee II on Cellulose (E. S. McColley, chairman) prepared the three proposed tentative methods of test referred to earlier in this report.

The Task Group on Pentosans has tested and approved a colorimetric procedure based on the Swedish orcinol method. The Task Group on Functional Groups is studying procedures for carboxyl and carbonyl. A proposed procedure for extractives, using 1:2, alcohol:benzene is under consideration.

Subcommittee III on Organic Esters (L. B. Genung, chairman) is engaged in revising Method D 871 to include new test procedures for acetyl (alternate), hydroxyl, intrinsic viscosity, color, haze, combined sulfur, and heat stability.

Subcommittee IV on Inorganic Esters (C. B. Gilbert, chairman) has been notified that Committee D-1 on Paint, Varnish, Lacquer, and Related Products has accepted tentatively the single method for the determination of the dilution ratios of cellulose nitrate, solvents, and diluents which was submitted to them. Method D 301, which is under Committee D-23 jurisdiction, will be revised accordingly.

Subcommittee V on Cellulose Ethers (R. W. Swinehart, chairman).—The Task Group on Purified Sodium Carboxymethylcellulose is continuing laboratory work on this material. Also work is continuing on preparing methods for hydroxyethyl cellulose.

Subcommittee VI on Statistics (D. O. Yandle, chairman).—The chairman is available for consultation on programs for interlaboratory testing and other statistical work.

This report has been submitted to letter ballot of the committee, which consists of 45 voting members; 24 have returned their ballots, of whom 20 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. A. SIMMONDS,
Chairman.

W. W. BECKER,
Secretary.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-23 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Pentosans in Cellulose (Orcinol Colorimetric Method) (D 1787 - 60 T).

Withdrawal of Tentative Method of Test for:

Pentosans in Cellulose (Aniline Acetate Colorimetric Method) (D 1438 - 56 T).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the new tentative method appears in the 1960 Supplement to Book of ASTM Standards, Part 6.

REPORT OF COMMITTEE D-24

ON

CARBON BLACK*

Committee D-24 on Carbon Black and its subcommittees held two meetings during the year: in Atlantic City, N. J., on June 25, 1959, and in Chicago, Ill., on Feb. 3, 1960.

J. H. Gifford has succeeded J. A. Tallant as chairman of Subcommittee IV on Nomenclature. A new Subcommittee VII on Specifications was organized and J. H. Gifford was appointed its chairman. The new subcommittee will coordinate its activities with those of Subcommittee XXIX on Compounding Ingredients of Committee D-11 on Rubber and Rubber-like Materials. The functions of Subcommittee VI on Coordination with Other Committees have been transferred to the Advisory Committee and the subcommittee was discontinued.

During the year the committee was unfortunate in losing through death the valued counsel and friendship of S. R. Doner who had been its first chairman.

The committee consists of 48 members, of whom 12 are classified as producers, 28 as consumers, and 8 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, N. P. Bekema.
Vice-Chairman, J. F. Svetlik.
Secretary, T. D. Bolt.

NEW TENTATIVES

The committee recommends for publication as tentative, jointly with Committee D-11 on Rubber and Rubber-like Materials, the following method and specification as appended hereto:[1]

Tentative Specifications for:
Carbon Blacks Used in Rubber Products.

Tentative Method for:
Sampling Packaged Shipments of Carbon Black.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:
pH Value of Carbon Black (**D 1512 – 57 T**),[2]
Pour Density of Pelleted Carbon Black (**D 1513 – 57 T**),[2]
Sieve Residue from Carbon Black (**D 1514 – 57 T**),[2] and
Sulfur Content of Carbon Black (**D 1619 – 58 T**).[2]

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Method of Test for Fines Content of Pelleted Carbon Black (D 1508 – 57 T):[2]

Footnote 3.—Add a sentence to read as follows: "Refer to ASTM Method D 1511 for a description of the apparatus."

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

[2] 1958 Book of ASTM Standards, Part 9.

Section 3(e).—Change to read: "Allow the sieve assembly to shake for the specified time."

Section 5.—Change Items (*1*) and (*2*) to Paragraphs (*a*) and (*b*) and add a new Paragraph (*c*) to read as follows: "(*c*) Running time of the test with the hammer operating."

Tentative Method of Test for Iodine Adsorption Number of Carbon Black (D 1510 – 57 T):[2] Revise as appended hereto.[3]

Tentative Method of Test for Pellet Size Distribution of Carbon Black (D 1511 – 57 T):[2]

Section 2(a).—Delete and substitute the following:

> (*a*) *Ro-Tap Sieve Shaker.*—This is a mechanically operated sieve shaker which imparts a uniform rotary and tapping motion to a stack of 8-in. sieves as described in Section 2(*b*). The Ro-Tap should be adjusted to accommodate a stack of five sieves, receiver pan, and cover plate. The bottom stops should be adjusted to give a clearance of 0.061-in. between the bottom plate and the screens so that the screens will be free to rotate. The Ro-Tap machine shall be powered with a ¼-hp electric motor producing 1725 to 1750 rpm. This will produce 140 to 160 raps per min and 280 to 320 rotary motions per min. The cover plate on the Ro-Tap shall be fitted with a cork stopper which shall extend from ⅛ to ⅜ in. above the metal recess. At no time shall a rubber, wood, or other material other than cork be permitted.

Section 3(c).—At the end of the sentence add the phrase: "with the hammer operating."

Footnotes 3 and 5.—Delete, renumbering remaining footnotes accordingly.

Section 5.—Delete Item (*2*) and designate Items (*1*) and (*3*) as (*a*) and (*b*).

Tentative Method of Test for Volatile Content of Carbon Black (D 1620 – 58 T):[2]

Section 1.—Add the following new sentence: "The volatile content test applies more specifically to those blacks used by the paint and ink industry."

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Method of Test for Attrition of Pelleted Carbon Black (**D 1507 – 57 T**).[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Physical Tests (J. F. Svetlik, chairman) has recommended that several tentatives under its jurisdiction be adopted as standard and is continuing work on a test method for mass strength of pelleted carbon black.

Subcommittee II on Chemical Tests (L. G. Mason, chairman) has recommended that several tentatives under its jurisdiction be adopted as standard, with some revisions for clarification.

Subcommittee III on Optical Tests (J. E. Smith, chairman) has considered modification of the benzene discoloration method (D 1618) and recommends continuation of the method as tentative without revision.

Subcommittee IV on Nomenclature (J. F. Gifford, chairman) has considered nomenclature on two types of carbon black and will consider methods of classifying rubber grade carbon blacks.

Subcommittee V on Sampling (R. O. Treat, chairman) recommended the Tentative Method for Sampling of Packaged Shipments of Carbon Black appended to this report, and is working on a method for sampling bulk shipments of carbon black.

---

[3] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 9.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VII on Specifications (J. F. Gifford, chairman) recommended the Tentative Specification for Carbon Blacks appended to this report.

This report has been submitted to letter ballot of the committee, which consists of 48 members; 33 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. P. BEKEMA,
Chairman.

T. D. BOLT,
Secretary.

REPORT OF COMMITTEE D-25

ON

CASEIN AND SIMILAR PROTEIN MATERIALS*

Committee D-25 on Casein and Similar Protein Materials held two meetings during the year: on June 24, 1959 in Atlantic City, N. J., and on Feb. 4, 1959 in Chicago, Ill. A meeting of Subcommittee IV, Section A on Paper Coating Tests, was held Feb. 24 in conjunction with the National TAPPI Convention.

D. A. Hillard of the Goodyear Tire and Rubber Co. was elected a member of the committee, representing Committee D-1 on Paint, Varnish, Lacquer, and Related Products, and Mark Jobson of the Perkins Glue Co. was elected to represent Committee D-14 on Adhesives. Other new members are E. T. McBroom of the Central Soya Co., J. R. Borst of the John R. Spellacy Dairy Products Co., and D. R. Zimmerman of S. C. Johnson & Son, Inc.

J. Rice of the Lowe Paper Co. resigned as representative of the TAPPI Coating Committee and also as chairman of Subcommittee II. J. F. Voit has been appointed the new representative of the TAPPI Coating Committee; a new chairman of Subcommittee II will be appointed. Ray Olson resigned as chairman of Subcommittee III and has been replaced by E. T. McBroom of the Central Soya Co.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. E. Clark.
Vice-Chairman, E. Maynard.
Secretary, L. E. Georgevits.
Advisory Committee: F. R. Adams and J. F. Voit.

At the present time, Committee D-25 consists of 30 members, of whom 23 are voting members; 7 are classified as producers, 7 as consumers, 9 as general interest members, with 30 consulting members.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Method of Test for:

Ash in Casein and Isolated Soy Protein,
Fixed Ash in Casein and Isolated Soy Protein,
Oil in Casein and Isolated Soy Protein,
Free Acidity in Casein and Isolated Soy Protein,
Moisture in Casein and Isolated Soy Protein,
Nitrogen in Casein and Isolated Soy Protein, and
Alkali Requrement of Casein and Isolated Soy Protein.

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Editorial and Nomenclature (M. E. Stonebreaker, chairman) will do more work in defining the various terms used by the committee. Some of the terms to be studied are: isolated soy protein, soy flour, mesh analysis, and particle size.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Sampling (Mr. J. C. Rice, chairman).—A proposed sampling has been issued as Tentative Method D 1697 - 59 T. No future work is planned by this subcommittee at present.

Subcommittee III on Chemical Tests (Mr. E. T. McBroom, chairman).—Test methods for fat in casein and isolated soy protein are being reviewed. It was proposed that an ether extraction be made on the residue of the Roese-Gottlieb test. New test methods on bacteria count are being studied.

Subcommittee IV on Physical Tests (Mr. E. Maynard, chairman).—Test methods for foam and dirt content are being revised and will be sent out to the subcommittee members for comment without further interlaboratory tests. Further interlaboratory tests are planned using the revised procedures for insolubles, minimum alkali, and odor.

Section A on Paper Coating Tests (L. E. Georgevits, chairman).—Samples of clay slurry, casein, isolated soy protein, and paper will be sent out to the members of this section. They will prepare paper coatings, check pH and viscosity, and make drawdowns. Each laboratory will send a set of drawdowns to the other laboratories and they will each test the complete set for wax pick. By this procedure it will be possible to determine if a laboratory is obtaining poor results on their drawdowns or wax pick results.

Section B on Sieve Analysis (Mr. J. Dore, chairman) has been set up to study the effect of humidity conditions on the sieve analysis of fine mesh caseins and soy protein. Tests will be made at 20 to 50 per cent and 90 per cent relative humidity.

Section C on Viscosity Tests (Mr. L. Clark, chairman) has been set up to study various instruments for measuring viscosity. Included will be the Brookfield viscometer, Stormer, Ford cup, Zein cup and Gardner tubes. Samples of two caseins differing widely in viscosity will be tested by two laboratories using the above instruments.

Subcommittee VI on Research (Mr. L. E. Clark, chairman).—No active projects have been set up at this time. A suggestion was made that tests should be considered to correlate more closely with the properties of casein and isolated soy protein with their performance in a joint cement.

This report has been submitted to letter ballot of the committee, which consists of 23 members; 16 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. W. Shader,
Chairman.

L. E. Georgevits,
Secretary.

REPORT OF COMMITTEE D-26

ON

HALOGENATED ORGANIC SOLVENTS*

Committee D-26 on Halogenated Organic Solvents held two meetings during the year: on June 25, 1959, at Atlantic City, N. J.; and on February 4 and 5, 1960, at Chicago, Ill., during ASTM Committee Week.

Following up previous organizational meetings, Committee D-26 organized its five subcommittees into smaller task groups with assigned problems. Several task groups have since met to expedite their work, and reports received indicate that specific advancements have been made so that tentative specifications for the assigned materials will soon be presented to the Society for approval. The problems proved to have broader implications than originally understood, and these must be investigated to avoid overlapping interests of other ASTM committees.

The importance of task group assignments and the holding of interim meetings has been stressed; the following of this schedule should result in tangible offerings by the June 1961 Annual Meeting.

The committee now consists of 100 members, of whom 47 are classified as producers, 33 as consumers, and 20 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. D. McMaster.
Vice-Chairman, G. M. Ford.
Secretary, Wanda L. Campbell.

Respectfully submitted on behalf of the committee,

V. E. Anspacher,
Chairman.

Wanda L. Campbell,
Secretary.

---

* Sixty-third Annual Meeting of the Society June 26–July 1, 1960.

REPORT OF COMMITTEE D-27

ON

ELECTRICAL INSULATING LIQUIDS AND GASES*

Committee D-27 on Electrical Insulating Liquids and Gases held three meetings during the past year: in Atlantic City, N. J., during the Annual Meeting of the Society, on June 22–24, 1959; in New York City on Nov. 18–20, 1959; and in Washington, D. C. on Feb. 22–24, 1960.

At the time of the Washington meeting the membership comprised 103, of whom 28 were classified as producers, 57 as consumers, and 18 as general interest members.

In connection with the fall 1959 meeting in New York, a symposium was held under the joint auspices of the Insulated Conductors Committee of the AIEE and Committee D-27 on the subject of High-Voltage Cable Insulation. Arrangements were made by V. R. Mulhall on behalf of Committee D-27. The symposium was attended by approximately 175 people from both organizations. Three papers were presented and discussed:

"Public Utility View of Cable Oils" by R. W. Gillette, of the Consolidated Edison Co. of New York, and A. M. Gates, of the Philadelphia Electric Co.

"Paper For High Voltage Cable" by W. A. Del-Mar, of the Phelps Dodge Copper Products Corp.

"Why and How of Cable Oil Tests" by R. B. Blodgett of the Okonite Co.

The papers and discussions will be made available as an ASTM Special Technical Publication *STP No. 253.*

The officers elected for the ensuing term of two years are as follows:

Chairman, E. R. Thomas.
Vice-Chairman, H. W. McCulloch, Jr.
Recording Secretary, C. A. Johnson.
Membership Secretary, R. M. Frey.

During the past year additional liaison representatives were appointed as follows:

J. C. Parker as representative of Committee D-27 on Committee D-9 on Electrical Insulating Materials.

F. M. Clark, as representative of Committee D-27 on the International Electrotechnical Commission.

G. L. Shombert, Jr. as representative of Committee D-27 on Committee E-11 on Quality Control of Materials.

E. L. Raab to continue as representative on the Transformer Committee on Askarels of AIEE.

J. C. Parker as a representative on the Switchgear Committee of the AIEE.

W. T. Gunn of Committee D-2 on Petroleum Products and Lubricants has been accepted as a liaison member of Committee D-27 representing Committee D-2.

Committee D-27 will lose one of its oldest members in the retirement of the chairman, Frank M. Clark, after the Annual Meeting. Mr. Clark had been a member of Subcommittee IV since 1924, becoming chairman in 1951, and taking a prominent part in the organization of Committee D-27 in 1958, of which he has served as chairman during the past two years.

Several publications of general interest in the preparation of which members of the committee have taken an active part are now available or will be ready for distribution in the future:

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

1. The questionnaire on Transformer Oil, circulated by the AIEE, will be published in summary form as a special technical bulletin, and will contain the results from 96 companies representing generating capacity of 70,000,000 kw. The tests to which the utilities attach importance fall in this order: (1) dielectric strength, (2) acidity, (3) power factor (dissipation factor), (4) interfacial tension, and (5) color.

2. A guide for the Maintenance of Transformer Askarels has been issued by the AIEE as *Publication No. 76.*

3. A Guide for Transformer Oil Practice has now been completed. It includes the suggested revisions which have been received during the first two years. It can be obtained from AIEE Headquarters in New York.

Research Reports File.—The results of the round robin on the Evaluation of Modified VDE Test for Determining the Dielectric Strength of Transformer Oil have been submitted to ASTM Headquarters for permanent file in the Research Reports File. The number assigned to this report is RR3:D-27. Any future reference to this material should use this designation.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee D-27 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Scavenger Content of Askarels (D 1701 – 59 T), and
Visual Examination of Used Askarels in the Field (D 1702 – 59 T).

Revision of Tentative Method of Test for:

Water in Insulating Oils by Extraction (D 1315 – 54 T).

These recommendations were accepted by the Standards Committee on September 15, 1959 and the new and revised tentative methods appear in the 1959 Supplement to Book of ASTM Standards, Part 9.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and recommended practice as appended hereto:[1]

Tentative Recommended Practice for:

Estimating Relative Temperature Limits of Dielectric Liquids.

Tentative Methods of Test for:

Refractive Index and Specific Optical Dispersion of Hydrocarbon Liquids,
Refractive Index of Askarels,
Sampling Askarels,
Volume of Oil in Oil-Contaminated Askarels,
Specific Gravity of Askarels, and
Free Chlorides in Askarels.

The last five new tentatives currently appear as separate methods in the Standard Methods of Testing Askarels (**D 901 – 56**).[2] The preparation of the askarel methods under separate headings is the first step in the endeavor to make Methods D 901 a collection of references to separate methods, similar to Methods D 117 for Transformer Oils. The sections of Methods D 901 where these tests currently appear are indicated below. In several cases it has been necessary to make minor changes in the methods in order to complete them for publication as separate standards; these changes are indicated below:

---

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 9.

[2] 1958 Book of ASTM Standards, Part 9.

Tentative Method of Test for Refractive Index of Askarels (present Sections 52 to 55 of Methods D 901):

New Section.—Add a new Section entitled Scope, to read as follows: "*Scope.*—This method of test covers a procedure for determining the refractive index of askarels."

Tentative Method for Sampling Askarels (present Sections 2 to 16 of Methods D 901)

Tentative Method of Test for Volume of Oil in Oil-Contaminated Askarels (present Sections 46 to 51 of Methods D 901)

Tentative Method of Test for Specific Gravity of Askarels (present Sections 17 to 21 of Methods D 901):

New Section.—Add the following new section entitled Scope: "*Scope.*—This method of test covers a procedure for determining the specific gravity of askarels."

Tentative Method of Test for Free Chlorides in Askarels (present Sections 39 to 45 of Methods D 901):

New Section.—Add the following new section entitled Scope: "*Scope.*—This method of test covers a procedure for determining the free chloride content of askarels."

Adoption of Tentatives as Standard Without Revision

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

2,6-Ditertiary-Butyl Para-Cresol in New Electrical Insulating Oils (**D 1473 - 58 T**),[2]

Water in Insulating Liquids (Karl Fischer Method) (**D 1533 - 58 T**),[2] and

Peroxide Number of Mineral Insulating Oils (**D 1563 - 58 T**).[2]

Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Testing Askarels (**D 901 - 56**)[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 1.—Add the following at the end of Section 1 (before the note): "The test methods included are as listed in Table I."

New Table.—Add new Table I to read as shown in the accompanying Table I, renumbering subsequent tables accordingly.

Sections 2 to 16.—Replace Sections 2 to 16 with the following new Section 2:

TABLE I.—LIST OF TEST METHODS.
(New Table I for Methods D 901)

Test Method	Section	ASTM Designation of Test Method
Sampling	2	D 1809
Specific Gravity	3	D 1810
Color	4 to 8	
Viscosity	9 to 11	
Fire Point	12 to 14	
Pour Point	15 to 17	
Neutralization Number	18 to 20	
Free Chlorides	21	D 1811
Volume of Oil	22	D 1808
Refractive Index	23	D 1806
Power Factor and Dielectric Constant	24 to 26	
Dielectric Strength	27 to 30	
Scavenger Content	31	D 1701
Visual Examination of Used Askarels in the Field	32	D 1702

"2. *Procedure.*—Sampling of askarels shall be in accordance with the Method for Sampling Askarels (ASTM Designation: D 1809)."

Sections 17 to 21.—Replace Sections 17 to 21 with the following new Section 3: "3. *Procedure.*—The specific gravity of askarels shall be determined in accordance with the Method of Test for Specific Gravity of Askarels (ASTM Designation: D 1810)."

Sections 22 to 38.—Renumber as Sections 4 to 20.

Sections 39 to 45.—Replace Sections 39 to 45 with the following new Section

21: "21. *Procedure.*—The free chloride content of askarels shall be determined in accordance with the Method of Test for Free Chlorides in Askarels (ASTM Designation: D 1811)."

Sections 46 to 51.—Replace Sections 46 to 51 with the following new Section 22: "22. *Procedure.*—The volume of oil in oil-contaminated askarels shall be determined in accordance with the Method of Test for Volume of Oil in Oil-Contaminated Askarels (ASTM Designation: D 1808)."

Sections 52 to 55.—Replace Sections 52 to 55 with the following new Section 23: "23. *Procedure.*—The refractive index of askarels shall be determined in accordance with the Method of Test for Refractive Index of Askarels (ASTM Designation: D 1806)."

Sections 56 to 62.—Renumber as Sections 24 to 30.

New Sections.—Add new Sections 31 and 32 to read as follows:

SCAVENGER CONTENT

31. *Procedure.*—The scavenger content of askarels shall be determined in accordance with the Method of Test for Scavenger Content of Askarels (ASTM Designation: D 1701).

VISUAL EXAMINATION OF USED ASKARELS IN THE FIELD

32. *Procedure.*—Visual examination of used askarels in the field shall be in accordance with the Method of Test for Visual Examination of Used Askarels in the Field (ASTM Designation: D 1702).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Sludge Formation in Mineral Transformer Oil by Sludge Accumulation (**D 1314 – 54 T**),

Approximate Acidity of Used Electrical Insulating Oil of Petroleum Origin (**D 1534 – 58 T**), and

Scavenger Content of Askarels (**D 1701 – 59 T**).

Tentative Recommended Practice for:

The Purchase of Uninhibited Mineral Oil for Use in Transformers and in Oil Circuit Breakers (**D 1040 – 58 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Materials Subcommittee A on Mineral Oils (E. L. Raab, chairman) is a discussion group on future trends and changes in requirements. Developments in the International Electrotechnical Commission are reported by F. M. Clark. Specifications for light and heavy cable oils are still under discussion, but many controversial points have been solved. Many cable oil test methods are being adapted for capacitor oil evaluation. Switch oil test methods appear to require several new means of determining properties not essential in other insulating oils.

Materials Subcommittee B on Synthetics (T. K. Sloat, chairman) has been working with two different classes of liquids—askarels and silicones.

Methods D 901 on Askarels are being revised so as to contain only the definition and significance of the various tests with references to the appropriate standard method. This has meant that methods such as refractive index, sampling, specific gravity, free chlorides, and volume of oil in oil-contaminated askarel are being made into separate standard methods, thus withdrawing them from Methods D 901 and leaving only the reference to the methods. Methods D 1701 and D 1702 will also be listed in Methods D 901. Other characteristics of askarels are under study such as hydrolytic stability and potentiometric method for free chlorides.

The initial organization of the working group on silicone fluids is complete, and it is correlating the many methods being used by the silicone fluid manufacturers in their studies of these materials. It is from these tests that the standard methods will be developed.

---

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Materials Subcommittee C on Gases (R. B. Blodgett, chairman) was organized in June 1959. It has 32 members, an advisory section, and two working sections: Section I on Non-Halogenated Gases and Section II on Halogenated Gases (including certain volatile liquids). The subcommittee is formulating specifications for nitrogen in electrical apparatus. In the field of halogenated gases (including certain volatile liquids) it is surveying properties relating to purity, specifications, and acceptance analyses of gases and certain volatile liquids and engineering applications. It has recommended to the appropriate Methods Subcommittee the development of methods for dew point and vapor pressure of various gases.

Methods Subcommittee N on Electrical Tests (H. O. Bennett, chairman) is bringing out a tentative method for dielectric strength by a modified VDE apparatus which may become a real research tool. Consideration is being given to an oxidation test involving measurements of power factor as a means of evaluating the oxidation resistance of the oil. A start has been made on a method for estimating the dielectric strength of insulating gases. The modified Perilli gas evolution test is being used as a basis for comparing the gas-absorbing properties of cable oils. New tests are under way for determining the electric stability of cable oil and of combinations of oil and paper.

Methods Subcommittee O on Chemical Tests (H. W. McCulloch, Jr., chairman) has reviewed the cooperative work on the many oxidation tests that have been studied. In addition to the ASTM bomb sludge and sludge by accumulation methods, the compilation of ASTM Standards on Electrical Insulating Liquids (Dec. 1959) contains suggested procedures for oxidation characteristics and oxygen stability of transformer oils. A translation of the IEC sludge accumulation method has recently been circulated in Committee D-27. A power-factor valued oxidation test and a rotary bomb method will be studied.

Major subcommittee and section activity has been directed toward extension of the applicability of present methods to used insulating oil and other insulating liquids, such as askarels. The new Method D 1701, Scavenger Content of Askarels, has been accepted by the Society since the 1959 Annual Meeting. Section work has been completed and balloting initiated on a proposed method for the determination of hydrolyzable chlorine compounds in askarels. The Recommended Practice for Estimating Temperature Limits on Dielectric Liquids is being recommended as a new tentative, as noted above. Work is under way to determine the additive content in service-aged inhibited oils, while a new section has been formed to study the stability of askarels.

Methods Subcommittee P on Physical Tests (J. C. Parker, chairman) has completed several new test methods during the past year. A method for the visual examination of used askarel (D 1702) is now available, and a method for the refractive index and specific optical dispersion values is appended to this report.[1] A new method for gas content of oil is being offered for letter ballot approval, while revision of the methods for water by extraction, and sampling, have already been accepted. Sections are being organized to develop a method for determining the dew point of gases, the volatility of insulating liquids, and the vapor pressure of halogenated gas. Work is continuing on the study of emulsion tests, an infrared test, and test for coefficient of thermal expansion.

This report has been submitted to letter ballot of the committee, which consists of 106 members, of whom 76 are voting representatives; 56 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

FRANK M. CLARK,
Chairman.

CHARLES A. JOHNSON,
Recording Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-27 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Dielectric Strength of Insulating Oil of Petroleum Origin (Using VDE Electrodes) (D 1816 - 60 T),

Hydrolyzable Chlorine Compounds in Chlorinated Aromatic Hydrocarbons (Askarels) (D 1820 - 60 T), and

Inorganic Chlorides in Askarels (D 1821 - 60 T).

New Tentative Continuity Specifications for:

Electrical Insulating Oil for Low-Pressure Cable Systems (D 1818 - 60 T), and

Electrical Insulating Oil for High-Pressure Cable Systems (D 1819 - 60 T).

The new Tentative Method D 1816 was accepted by the Standards Committee on September 29, 1960, and appears in the 1960 Supplement to Book of ASTM Standards, Part 9. The other four new tentatives were accepted on December 29, 1960, and are available as separate reprints.

REPORT OF ALCA-ASTM JOINT COMMITTEE ON LEATHER*

The ALCA-ASTM Committee on Leather (joint with the American Leather Chemists' Assn.) held two meetings during the year: on Nov. 12 and 13, 1959 and March 17 and 18, 1960. Both meetings were held in Philadelphia, Pa.

The officers elected for the ensuing term of two years are as follows:

Chairman, Joseph Naghski.
Vice-Chairman, B. L. Lewis.
Secretary, Milton Bailey.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Proposed Tentative Method for:

Measuring Thickness of Leather Units,
Measuring Thickness of Leather Test Specimens, and
Test for Water Absorption (Static) of Leather.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Method of Test for Bond Strength of Leather Belting (**D 1699 – 59 T**)[2] and continuation of the method as tentative:

Section 2(b).—In the last sentence insert the word "not" so that the sentcnee reads: "The nature of the frequency distribution of the data secured by this method cannot . . . ."

Section 5(a).—To the end of the sentence add the words: "and test immediately while wet."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of:

Test for Area of Leather (**D 1515–57 T**),[3]
Test for Width of Leather (**D 1516–57 T**),[3]
Test for Corrosion Produced by Leather in Contact with Metal (**D 1611–58 T**),[3] and
Conditioning Leather and Leather Products for Testing (**D 1610–58 T**).[3]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Definitions of Terms Relating to Leather (**D 1517 – 57 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

New Definitions.—Add the following new definitions in their proper alphabetical sequence:

Water Repellency.—The ability of a leather surface to resist wetting by liquid water.

Water Resistance.—The ability of a leather to resist absorption and transmission of liquid water.

Waterproofness.—Non-transmission of liquid water through the cross-section of the leather.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 6.

[2] 1959 Supplement to Book of ASTM Standards, Part 6.

[3] 1958 Book of ASTM Standards, Part 6.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

A listing is given under the appropriate subcommittee or section of all assigned methods, together with their present ALCA and ASTM designations. A long term objective of the committee is to establish all the ALCA methods, possibly in some modified form, as joint methods approved by ASTM. The progress to date is indicated by those methods that have both ALCA and ASTM designations.

Subcommittee I on Nomenclature, Definitions, and Applicability (R. M. Lollar, chairman):

	ASTM Designation
Definition of Terms Relating to Leather	D 1517 – 57 T

The subcommittee reviews test methods with regard to definitions and applicability. Particular attention is being given to statements of significance and precision of all methods being worked on by the committee. The subcommittee recommended the adoption of definitions for water repellency, water resistance, and waterproofness, as noted earlier in this report.

Subcommittee II on Sampling and Conditioning for Physical Testing (C. W. Mann, chairman):

Methods	ALCA Designation	ASTM Designation
Method of Conditioning Leather and Leather Products for Testing	E1	D 1610 – 58 T
Sampling Light Leather for Physical Tests	J1	...
Sampling Heavy Leather for Physical Tests	J2	...
Sampling Small Skins for Physical Tests	...	...

Methods for sampling cattlehide side leather and small skins are being prepared. A sampling method for research and development test is being prepared for review.

Subcommittee III on Physical Testing (G. A. Butz, chairman):

Section 2 on Physical Dimensions (J. Naghski, chairman):

Methods	ALCA Designation	ASTM Designation
Area of Leather	E2, E2a	D 1515 – 57 T
Width of Leather	E5	D 1516 – 57 T
Thickness of Leather Units	E3	...
Thickness of Leather Test Specimens	E4	...

Methods E3, Thickness of Leather Units, and E4, Thickness of Leather Test Specimens, have been revised and are being recommended to the Society for publication as tentative as noted earlier in this report.

Section 3 on Tensile Properties (R. L. Young, chairman):

Methods	ALCA Designation
Tongue Tear Strength	E10
Buckle Tear Strength	E11
Stitch Tear Strength, Single Hole	E12
Stitch Tear Strength, Double Hole	E13
Bursting Strength, Ball	E14
Tensile Strength	E15
Breaking Strength	E16
Elongation	E17
Alternate Method for Elongation	E17a
Slit Tear Resistance	...
Resistance to Grain Cracking (Mullen Test)	...

Methods for ball bursting strength, tensile strength, breaking strength, and elongation are being reviewed and revised in accordance with suggested changes. Modifications have been made in the procedures for the stitch tear tests and the slit tear resistance test. Procedures are being planned to determine the variability of the methods prior to recommendation to ASTM for publication as tentative.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Section 4 on Measurement of Water Resistance (R. G. Ashcraft, chairman):

Methods	ALCA Designation
Water Absorption (Static)	E30
Water Resistance (Dynamic)	E31
Permeability to Water Vapor	E32

Studies have been made with the Kubelka method for static water absorption. The error was found to be rather high and it was recommended that it be compared with the ALCA static test. Definitions for water repellency, water resistance, and waterproofness were approved. Significance and precision statements for the water resistance (dynamic) test method were approved. The proposed Tentative Method for Water Absorption of (Static) Leather appended to this report was developed by this section.

Section 5 on Surface Characteristics (Robert Stubbings, chairman):

Methods	ALCA Designation
Resistance to Cracking at Low Temperatures	E42
Staining	E44
Crocking	E46
Fire Resistance of Leather	E50

During the year work was completed on the method for flex testing of finish on upholstery leather. A method for cold-crack resistance of upholstery leather is being reviewed in the light of negative votes in the committee.

Laboratory and field test work on a large-scale test of lightfastness methods has been completed, and results are in the hands of the committee for evaluation. Laboratory work is under way on methods for determining crock resistance of finish in leather, scuff resistance of leather, and bleed resistance of leather.

Section 7 on Mechanical Leathers (A. Hirsch, chairman):

Methods	ASTM Designation
Test for Corrosion Produced by Leather in Contact with Metal	D 1611 - 58 T
Test for Bond Strength of Leather Belting	D 1699 - 59 T

Studies are under way on a proposed method to determine area stability of leather on immersion in water and oil. Three interlaboratory test series are being carried out following the proposed procedure in order to determine its reliability. Results of two series are complete, and those of the third series are expected shortly. It has been decided to run additional tests using prolonged exposure times at the temperatures which are suggested in the proposed method.

A revision in the Tentative Method of Test for Bond Strength of Leather Belting (D 1699) is being recommended to assure that the test is performed while the leather is still wet.

Section 8 on Deterioration (C. F. Dudley, chairman).—No ALCA or ASTM methods are presently under the jurisdiction of this section.

The work of the section is divided into four projects: Perspiration, Heat Stability, Dry Cleaning, and Laundering.

Two different methods to determine resistance to perspiration were evaluated in interlaboratory tests. Satisfactory data from the investigation will be used in the preparation of a test method.

Data have been obtained on an accelerated moist-tropical exposure at 60 C and 100 per cent RH for 1 and 2 weeks. These test conditions did not discriminate between the stability of straight chrome and chrome-vegetable retan leathers. Available information would indicate that such a discrimination would be necessary to make these conditions valid for an accelerated test. Two of the leathers, straight chrome and stuffed chrome-vegetable retan, will be evaluated at 40 C and 100 per cent RH for 1 and 2 weeks. Two determinations will be made, one with the leather at normal moisture content, the other after drumming the leather in water.

A method of test for resistance to dry cleaning is being studied in two laboratories. Temperatures of 70 and 90 F will be used, with the leather being conditioned prior to test at 50 and 90 per cent RH. The Launder-Ometer and the AATCC Tumbling Jar will be used. The resistance of the water repellency properties of the leather to this treatment will be studied by means of the spray rating.

Data from an interlaboratory test on laundering have been obtained. The resistance of the leather to the test was evaluated by colorfastness, area retention, and temper retention. Because of the wide variability in the responses to these tests, it was decided that studies should be made on the methods used in the evaluation. A method for measuring colorfastness will be studied first.

Section 10 on Miscellaneous Physical Properties (Milton Bailey, chairman):

Methods	ALCA Designation
Grain Cracking (Heavy Leather)	E41
Stiffness	E43
Compressibility of Leather	E45
Piping	E40

Studies are under way on methods for wet abrasion, piping, compressibility, and break of leather. Data on the precision and accuracy of the method for compressibility were rejected because of high interlaboratory variations. Additional data are being obtained. The method used for wet abrasion showed significant differences between pre- and post-treated leathers. The section was assigned the task of developing methods to determine the electrical conductivity of leather.

Subcommittee IV on Research (M. M. Baldwin, chairman).—No methods are assigned to this subcommittee. The objectives of the committee are to study pertinent research work, to survey the area between research and test method, and to recommend the development of test methods.

The committee is surveying methods relative to the comfort of leather such as water vapor permeability and water absorption, and is considering the type of factors involved in the psychophysical testing of leather.

It is also engaged in the consideration of all types of nondestructive tests as a continual function. The development of an electrical conductivity test for leather has, through the Advisory Committee, been referred to the Section on Miscellaneous Properties. A method on heat conductivity has been investigated and interlaboratory tests are planned. The results are promising and indicate that a suitable method can be established.

The subcommittee recommends suitable subjects among those under study by the committee for formal discussion during meetings of the main committee.

Subcommittee V on Editorial (T. C. Thorstensen, chairman) has concerned itself principally with the form and organization for presentation of test methods.

This report has been submitted to letter ballot of the committee, which consists of 67 members; 30 members returned their ballots, of whom 27 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. KANAGY,
Chairman.

C. W. MANN,
Secretary.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING*

Committee E-1 on Methods of Testing held two meetings during the year: in Philadelphia, Pa. on Dec. 1, 1959 and April 26, 1960. Twenty-nine subcommittees and task groups met during the 1959 Annual Meeting of the Society in Atlantic City. Subcommittee 17 on Thermometers met in Chicago on Dec. 9, 1959. Meetings were held of 20 subcommittees and task groups during ASTM Committee Week in Chicago, Ill., from Feb. 1–5, 1960. Subcommittee 28 on Microscopy and several of its task groups met in New York City on March 10, 1960.

The following three members of Committee E-1 were appointed to the committee during the year by the Board of Directors: Lyman Fourt, Harris Research Laboratories, Inc., 3-year term; N. C. Schultz, Olin Mathieson Chemical Corp., 3-year term; and Howard Tangenberg, The B. F. Goodrich Co., 2-year term. In addition, Messrs. A. C. Webber and H. A. Birdsall were each reappointed for a 3-year term.

The committee records with sincere sorrow the passing of Mr. H. F. Moore on April 16, 1960, at the age of 85 years. Mr. Moore had a distinguished record of long and active service in the Society since 1903 and was an active member of Committee E-1 for 44 years. He was an Honorary Member of the committee since 1952. He will be missed by his many friends in the Society and in Committee E-1 who held him in deep affection and high esteem.

Two new task groups are being organized by Committee E-1 during the 1960 Annual Meeting: one on Thermal Conductivity Testing of Metals under the chairmanship of Mr. U. U. Savolainen, Texas Instruments, Inc., and the other on Linear Expansion Test with H. E. Hagy of Corning Glass Works as chairman.

The activities of the committee dealing with methods for the determination of water, and correlation of methods for chemical analysis have been transferred to Committee E-15 on Analysis and Testing of Industrial Chemicals. Accordingly, Subcommittee 13 on Determination of Water and Subcommittee 15 on Correlation of Chemical Analysis have been discontinued.

Subcommittee 24 on Cupping Tests has been discontinued due to lack of interest in continuing work on this subject.

Committee E-1 is sponsoring a Symposium on Shear and Torsion Testing during the 1960 Annual Meeting of the Society.[1]

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee E-1 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The papers in this Symposium have been published as *ASTM STP No. 289.*

New Tentative Specifications for:

Apparatus for Microdetermination of Nitrogen by Kjeldahl Method (E 147 – 59 T), and

Apparatus for the Microdetermination of Nitrogen by the Dumas Method (E 148 – 59 T).

New Tentative Method of Test for:

Bulk Density of Abrasive Grains (E 153 – 59 T).

Proposed Revision of Tentative Methods of:

Compression Testing of Metallic Materials (E 9 – 52 T), and

Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 57 T).

The above recommendations were accepted by the Standards Committee on September 15 and October 2, 1959. These new and revised standards are published in the 1959 Supplement to Book of ASTM Standards, Parts 3, 5, and 7.

NEW TENTATIVE

The committee recommends for publication as tentative the Specifications for Precision Micromesh Sieves as appended hereto.[2]

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (E 11 – 58 T):[3] Revise as appended hereto.[4]

Tentative Specification for Apparatus for Determination of Water by Distillation (E 123 – 56 T):[5]

Section 1.—Add the following at the end of this section: "and other similar ASTM methods."

Section 2.—In the second sentence after "petroleum products" add "soaps, naval stores, etc."

Section 3.—Change to read as follows:

> 3. *Glass Flask.*—The glass flasks shall have nominal capacities of 500 ml or 1000 ml, as required, and shall be made of heat-resistant glass. Styles shall be as shown in Figs. 3 and 4, and as listed in the following table:

Style	Size	Shape	Top Finish	Figure
A	500 ml	.....Round	With \$ Joint	3
B	500 ml	.....Round	With \$ Joint	3
C	1000 ml	.....Round	With \$ Joint	3
D	500 ml	.....Erlenmeyer	With \$ Joint	4

Section 7.—Revise to read as follows:

> 7. *Traps.*—The traps shall be of 5-ml, 10-ml, and 25-ml capacities. Five styles are provided as specified in Table I and shown in Figs. 7, 8, 9, 10, and 11.

Table I.—Add a new Table I giving dimensions and sizes of traps, as shown in the accompanying Table I.[6]

Figure 3.—Add to Fig. 3 a third illustration "C" showing dimensions of the 1000-ml round-bottomed flask with standard taper 24/40 joint as shown in the accompanying Fig. 1.[6]

Change the title of Fig. 3 to read: "Glass Flasks, Round-bottomed." Revise the title of A to read: "500 ml with Taper Joint." Revise the title of B to read: "500 ml with Ball-and-Socket Joint." Add a title for C to read: "1000-ml with Taper Joint."

---

[2] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Parts 3, 7, 8, and 9.

[3] 1958 Book of ASTM Standards, Parts 3, 4, 5, 7, 8, 9, and 10.

[4] The revised tentative appears in the 1960 Supplement to Book of ASTM Standards, Parts 3, 4, 5, 7, 8, 9, and 10.

[5] 1958 Book of ASTM Standards, Parts 4, 7, and 8.

[6] The new table and figures are not included in this report but appear in the revised Specifications E 123; see 1960 Supplement to Book of ASTM Standards, Parts 4, 7, and 8.

New Fig. 4.—Add as a new Fig. 4 the accompanying Fig. 2[6] showing dimensions of the 500-ml Erlenmeyer flask with a standard taper 24/40 joint, renumbering the present figures accordingly.

New Fig. 11.—Add as new Fig. 11 the accompanying Fig. 3[6] showing 5-ml and 10-ml traps graduated in 0.1-ml subdivisions.

TENTATIVE REVISION OF STANDARD

The committee recommends that the six ASTM Solvent Distillation Thermometers shown in the accompanying Table II[7] and the Standardization Temperatures in Table III[7] be published as tentative as a tentative revision of the Standard Specifications for ASTM Thermometers (**E 1 - 59**).[8]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for ASTM Thermometers (E 1 - 59):[8]

Section 9.—Add the precision thermometers 69C, 69F, 70C and 70F to those requiring the test for bulb stability with the following test temperature range:

ASTM Thermometer No.	Bulb Stability Test Temperature Range
69C................	280 to 290 C
69F................	540 to 560 F
70C................	360 to 370 C
70F................	680 to 700 F

Table I, Thermometers 67 F and 68 F.—Change the range of the ice point scale from "31 to 33 F" to "30 to 34 F."

Thermometers 69 C and 70 C.—Change the range of the ice point scale from "−1 to + 1 C" to "−2 to + 2 C."

The above changes in the ice point scale will provide additional graduations either side of the ice point as now provided for other thermometers.

Thermometers 82F to 87F.—Add a tolerance of ± 2 mm to the distance from the bottom of the bulb to the bottom of the stem enlargement.

Standard Method of Determining Relative Humidity (D 337 - 34):[9] Revise as appended hereto.[10]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Methods for Notched Bar Impact Testing of Metallic Materials (**E 23 - 56 T**)[11] be approved for reference to letter ballot of the Society for adoption as standard, with revision as follows:

Section 27.—Change Paragraph (*a*) to read as follows:

> 27. (*a*) Calibration also will require inspection for the general requirements of Section 5, checking and adjustment if necessary of the various dimensions prescribed for the particular method of test in Sections 6 and 7 and other general precautions such as the following:

Omit Paragraph (*f*).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[12]

---

[7] The new tables are not included in this report but appear in the revised specifications E 1; see 1960 Supplement to Book of ASTM Standards, Parts 7 and 8.

[8] 1959 Supplement to Book of ASTM Standards, Part 7.

[9] 1958 Book of ASTM Standards, Part 10.

[10] The revised method appears in the 1960 Supplement to Book of ASTM Standards, Part 10.

[11] 1958 Book of ASTM Standards, Part 3.

[12] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman).—Consideration has been given to the possible need for revision of Methods of Verification of Testing Machines (E 4) to provide for the calibration of field-type testing equipment used for testing concrete and masonry products. It was decided that no action should be taken to revise the specification to permit greater errors for this type of equipment until the appropriate product committees request such action.

A task group composed of Messrs. A. A. Moore (chairman), F. M. Howell, W. H. Mayo, and D. R. Tate was appointed to study the question of needs for additional methods of calibration of testing machines at the high rates of straining now required by the Recommended Practices for Short-Time Elevated Temperature Tension Tests of Materials (E 21), and for Tension Tests of Metallic Materials at Elevated Temperatures with Rapid Strain Rates (E 151).

Several manufacturers are providing machines designed for load and strain recording in the speed range covered by these recommended practices. It is hoped that as these machines come into use, satisfactory methods of calibration will evolve. No single method of calibration used to date seemed satisfactory so the task group plans to follow developments in the field before recommending a method of calibration.

It was agreed, subject to letter ballot, that the time interval between verifications of testing machines in constant use be changed from 6 months to 12 months.[13]

A task group consisting of Messrs. R. H. Heyer (chairman), F. C. Huyser, R. P. Lathrop, and R. S. Strimel was appointed to consider whether the magnification ratio of an extensometer is adequately described in the Method of Verification and Classification of Extensometers (E 83) and to study the need for clarification of the definition of error.

Subcommittee 2 on Effect of Speed in Mechanical Testing (L. K. Irwin, chairman).—The Task Group on Speed Characteristics of Testing Machines (R. W. Fenn, Jr., chairman) presented graphical results of the strain-rate and load-rate capacities of four conventional testing machines. This information was compared with the requirements in ASTM Recommended Practice for Short-Time Elevated Temperature Tension Tests of Materials (E 21). The methods used to obtain the data, the per cent error between dynamic and static loading, changes in apparent modulus, *E*, and the performance of five microformer type strain records were discussed. The conclusions reached were that conventional testing machines are not satisfactory for all loading rates permitted by Recommended Practice for Tension Tests of Metallic Materials at Elevated Temperatures with Rapid Heating and Conventional or Rapid Strain Rates (E 151) and that a method for determining the maximum capabilities of existing machines should be developed.

At its February meeting the subcommittee discussed certain performance characteristics of new available commercial equipment. It is believed that at high rates of loading and straining automatic start-stop controls, load and strain recording systems may be required. The new problems in materials testing indicate the need for testing machines to be equipped with such devices.

Subcommittee 3 on Elastic Properties and Definitions on Mechanical Testing (G. R. Gohn, chairman).—The Task Group on Review of Definitions (G. R. Gohn, acting chairman) is preparing definitions to be added to the Tentative

---

[13] See Editorial Note, p. 446.

Definitions of Terms Relating to Methods of Mechanical Testing (E 6).[13] Corresponding editorial revisions will be made in the methods for Young's modulus (E 111) and shear modulus (E 143).

The Task Group on Elastic Constants (J. A. Miller, chairman) has recommended editorial changes in the Method for Determination of Young's Modulus (E 111) and Tentative Method of Test for Poisson's Ratio at Room Temperature (E 132) in keeping with a previous revision to the Method for Determination of Shear Modulus at Room Temperature (E 143).

The Task Group on Elastic Constants recommended for consideration as an "E" tentative method of test the proposed Tentative Method of Test for Static Modulus of Elasticity and Poisson's Ratio in Compression of Cylindrical Concrete Specimens prepared by Committee C-9 on Concrete and Concrete Aggregates. This method is applicable to testing brick, stone, and other materials and should be published as a general method of testing. The letter ballot of the subcommittee, however, developed several important suggestions which deserve further consideration.

The next project for the task group is to develop a method of test for the determination of Young's modulus at elevated temperatures for the range from ambient to 1000 F.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—The Joint ASTM-SAE Task Group on Hardness-Tensile Conversions for Steel has recommended changes in the hardness conversion tables now in use in the SAE Handbook, ASM Handbook, Federal Test Method 241.2, and ASTM Methods and Definitions for Mechanical Testing of Steel Products (A 370). Committee A-1 on Steel will consider appropriate changes in Methods A 370.

A table of corrections for Rockwell B tests on cylindrical surfaces was approved last year and has been incorporated in the Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18).

Knoop hardness conversions for Nickel and High-Nickel Alloys are being developed. The original test data are being checked by three additional laboratories.

Certain proposed changes in the Brinell hardness methods (E 10) and rapid indentation hardness methods (E 103) are now being voted upon by the subcommittee.[13] A proposed change in the Rockwell hardness methods (E 18) concerning calibration of Rockwell testers with test blocks is also the subject of subcommittee letter ballot.[13]

Subcommittee 7 on Impact Testing (W. W. Werring, chairman) has recommended the adoption as standard of the Tentative Methods E 23.

Subcommittee 9 on Rheological Properties (W. F. Fair, Jr., chairman).—The Task Group on Softening Point (M. D. Chamberlain, chairman) has been cooperating with Subcommittee B-5 of Committee D-4 on Road and Paving Materials in studying certain details of the Method of Test for Softening Point by Ring and Ball Apparatus (E 28).

The Task Group on Methods and Apparatus for Absolute Viscosity Measurements (W. F. Fair, Jr., chairman) has been considering the proposals submitted by the USSR on the absolute value of the viscosity of water. A statistical review of the data is to be made at the National Bureau of Standards.

Subcommittee 10 on Sieves (L. V. Judson, chairman) prepared the Specifications for Precision Micromesh Sieves being submitted as tentative. These specifications have resulted from the Symposium on Particle Size Measurement[14] held at the June, 1958, Annual Meeting.

---

[14] The papers in this symposium have been published as *ASTM STP No. 234*.

The revision of Specifications E 11 being submitted is the result of certain proposals discussed at the meeting of ISO/TC 24 on Sieves. Two members of the subcommittee, L. V. Judson and A. E. Reed, attended the ISO meetings as members of the American Delegation. A résumé of the accomplishments of the ISO meeting appears in *The Magazine of Standards*, January, 1960.

Subcommittee 11 on Sub-Sieve Testing (L. T. Work, chairman).—The Task Group on Sub-Sieve Testing by the Optical Microscope (R. P. Loveland, chairman) has in preparation methods for determining the concentration and average particle size by the count method. This will involve consideration of the basic microscopy and correlation of the count method with other particle size methods such as those for grain count and size frequency.

Programs of work are being developed by the Task Group on Choice of Test Methods (Mrs. Katharine Mather, chairman) and Task Group on Sedimentation Methods (K. T. Whitby, chairman).

Subcommittee 14 on Conditioning and Weathering (F. M. Gavan, chairman).—Revision of the Method for Determination of Relative Humidity (D 337) has now been completed and will be published as an "E" method.[13] The Tentative Definitions with Procedures Relating to Conditioning and Weathering (E 41) are being revised and separated into two separate tentatives; one will cover Nomenclature and Definitions Relating to Conditioning, and the other Specifications for Standard Atmosphere for Conditioning and Weathering.[13] Methods are in preparation for outdoor exposure and the operation of the enclosed carbon-arc; also specifications for laboratory testing enclosures other than ovens. Revision of the Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (E 96) is under consideration. Steps are being taken to develop the use of "Mylar" polyester film as a standard material for the evaluation of water-vapor transmission tests.

Subcommittee 16 on Thickness Measurement (S. B. Newman, chairman) is preparing a revision of the Methods of Test for Thickness of Solid Electrical Insulation (D 374) so that it may be applicable to a wider variety of engineering materials.

Subcommittee 17 on Thermometers (R. D. Thompson, chairman).—Specifications for a new set of distillation thermometers were developed at the request of Committee D-1 on Paint, Varnish, Lacquer, and Related Products. These are short-range, high-sensitivity instruments designed for testing materials of high purity having high temperature and relatively narrow boiling ranges. It is expected that these thermometers will be of value to a number of other technical committees working with materials of this type.

A new space-saving format for the Standard Specifications for ASTM Thermometers (E 1) was introduced last year. Whereas these specifications formerly required 51 pages in the Book of Standards, this has now been reduced to 34 pages. The new format provides greater convenience and simplicity for the user.

Active cooperation has continued with ISO/TC 48 on Laboratory Glassware and Related Apparatus in the development of precision thermometer specifications to serve as the basis of an international standard.

A number of projects are being actively pursued. These include a cooperative study at the National Bureau of Standards to compare the present ASTM method with a proposed ISO test for bulb stability, a revision of standardization temperatures, the development of specifications for distillation thermometers for materials of high but wide boiling range, and the development of two additional high-temperature Saybolt viscosity thermometers.

Subcommittee 18 on Hydrometers (R. D. Thompson, chairman).—Current projects include continued development of specifications for short-range, high-sensitivity instruments for use in testing liquified petroleum gases, and cooperative work with ISO/TC 28 and TC 48 to establish the best values for the expansion coefficient of hydrometer glasses used in calculating petroleum measurement tables.

Subcommittee 19 on Glassware Laboratory Apparatus (J. J. Moran, chairman) completed the revision of the Specifications for Apparatus for Determination of Water by Distillation (E 123) to include glassware apparatus used in determining water in naval stores and in soaps and other detergents. Specifications were also completed for two burets used for dilute tetraethyllead fluid used by Committee D-2 on Petroleum Products and Lubricants in engine test methods for rating motor fuels.

Subcommittee 21 on Metalware Laboratory Apparatus (W. J. Tancig, chairman).—The Task Group on Safety Requirements for Pressure Cylinders for Elastomer Aging (G. C. Maasen, chairman) met in Chicago on Feb. 3, 1960. It is presently comparing state regulations and insurance company recommendations for the operation of pressure vessels.

Subcommittee 25 on Shear and Torsion Tests (R. W. Fenn, Jr., chairman).—Arrangements for the Symposium on Shear and Torsion Testing[1] to be held during the 1960 Annual Meetings were developed by R. W. Fenn, Jr. (chairman), R. B. Clapper, and A. J. Herzog.

The Bibliography on Shear and Torsion Tests has been brought up to date by a task group under the chairmanship of Jerome Swanson.

The Torsion Test Task Group (J. Marin, chairman) is actively working on the establishment of a tentative torsion test practice. It is not certain when this material will be ready for subcommittee consideration.

A new task group or groups will be formed after the Symposium on Shear and Torsion Testing to proceed with the problem of preparing tentative recommended practices on shear testing.

Subcommittee 26 on Filtration Materials (T. L. King, chairman).—The Tentative Method of Test for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use (E 128) is being reviewed for possible revision and adoption as standard in 1961.

Subcommittee 27 on Low-Temperature Testing of Elastomers and Plastics (F. M. Gavan, chairman).—The second paper on rheological testing has been submitted for publication in the ASTM BULLETIN.[15] A paper being prepared by the Bureau of Ships of the Navy Department will be ready for coordination within the committee at the June, 1960, meeting. A paper on the correlation of the various low-temperature methods is still in preparation. The subcommittee is considering extending its scope to include high-temperature testing and the matter is being discussed with the interested committees.

Subcommittee 28 on Microscopy (T. G. Rochow, chairman).—The ASTM Bibliography on Microscopy is almost ready to be presented to Committee E-1. There is progress on specifications for microscopical slides and cover glasses, and also there is essential agreement on about 60 definitions of microscopical terms. A questionnaire on applications of and requirements for photomicrographical materials is being circulated within the subcommittee. The task of specifying procedures for polishing nonmetallic materials has been changed to recommended practices. A sixth task group has the assignment of naming and defining media for embedding specimens. Another informal Session on Non-commercial Microscopic Assessories is being arranged by Mr. M. C. Miller for the June, 1960, meeting.

---

[15] L. Boor, M. Hanok, F. Conant, and W. Scoville, Jr., "Rheological Testing of Elastomers at Low Temperatures," ASTM BULLETIN, No. 246, May, 1960, p. 25.

Subcommittee 29 on Microchemical Apparatus (A. Steyermark, chairman) completed during the year the new Specifications for Apparatus for Microdetermination of Nitrogen by Kjeldahl Method (E 147) and Specifications for Apparatus for Microdetermination of Nitrogen by Dumas Method (E 148). Consideration is now being given to apparatus used for micro-carbon and micro-hydrocarbon determinations.

Subcommittee 30 on Bend Test for Ductility (W. H. Mayo, chairman) has in preparation a guided bend test. It has also been cooperating with Committee A-1 on Steel in revision of the bend test procedures in Methods and Definitions for Mechanical Testing of Steel Products (A 370).

Subcommittee 32 on Thermocouples for Temperature Measurement (J. W. Freeman, chairman).—Since this subcommittee was organized in November 1958, it has been active through its five task groups on (1) Standardization of Standard Thermocouple Materials (D. I. Finch, chairman), (2) Newer Thermocouple Materials (A. I. Dahl, chairman), (3) Review of ASTM Methods (F. M. Gavan, chairman), (4) Monograph on Use of Thermocouples (J. W. Murdock, chairman), and (5) Atmospheres and Their Effect on Thermocouples (C. D. Starr, chairman). At its February meeting a new Task Group 6 on Application of Thermocouples for Airframe and Space Vehicles (R. B. Clapper, chairman) was authorized. This task group held its organization meeting in Los Angeles on May 12 and decided to consider a Symposium on the Application of Thermocouples in the Airframe and Space Industry.

This report has been submitted to letter ballot of the committee, which consists of 38 voting members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. WEBBER,
Chairman.

P. J. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Standard Atmospheres for Conditioning and Testing Materials (E 171 – 60 T).

Revision of Tentative Methods of:

Verification of Testing Machines (E 4 – 57 T),
Test for Brinell Hardness of Metallic Materials (E 10 – 58 T),
Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 59 T), and

Rapid Indentation Hardness Testing of Metallic Materials (E 103 – 54 T).

Revision of Tentative Definitions of:

Terms Relating to Methods of Mechanical Testing (E 6 – 58a T).

Revision of Tentative Definitions with Procedures Relating to:

Conditioning and Weathering (E 41 – 57 T), with a change in title to read, "Nomenclature and Definitions Relating to Conditioning."

Tentative Revision of Standard Hardness Conversion Tables for:

Metals (Relationship Between Brinell Hardness, Diamond Pyramid Hardness, Rockwell Hardness, and Rockwell Superficial Hardness) (E 140 – 58).

The revision of Tentative Definitions E 41 was accepted by the Standards Committee on December 19, 1960; the other recommendations were accepted on September 29, 1960. All appear in the 1960 Supplements to Book of ASTM Standards.

REPORT OF COMMITTEE E-2
ON
EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held three meetings during the year: in Atlantic City, N. J. on June 24, 1959; in San Francisco, Calif. on Oct. 12, 1959; and in Pittsburgh, Pa. on March 3, 1960. The Executive Subcommittee also met at these times.

A half-day Symposium on Spectroscopic Excitation was presented on June 24, 1959 at Atlantic City, N. J. during the Annual Meeting of the Society. It has since been published as *ASTM STP No. 259* (1960).

A three-day Symposium on Spectroscopy was held in cooperation with Committee E-13 on Absorption Spectroscopy during the Third Pacific Area National Meeting, San Francisco, Calif., October 13 through 16, 1959. The papers are to be published as *ASTM STP No. 269*.

The committee plans a third edition of the book, "Methods for Emission Spectrochemical Analysis," to be published in 1960.

The committee, in cooperation with Committee E-3 on Chemical Analysis of Metals, is planning a Symposium on Methods for Trace Analysis to be held at the Annual Meeting in Atlantic City, N. J. in June, 1961.

The committee consists of 204 members. This represents an increase of 4 since the last Annual Meeting. Several people from the area west of the Mississippi and from Canada have applied for membership.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. E. Michaelis.
Vice-Chairman, R. W. Smith.
Secretary, D. W. Henthorn.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee E-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Recommended Practices for:

Photographic Processing in Spectrochemical Analysis (**E 115 – 59 T**), and
Photographic Photometry in Spectrochemical Analysis (**E 116 – 59 T**).

Revision of Tentative Definitions of:

Terms and Symbols Relating to Emission Spectroscopy (**E 135 – 58 T**).

The revised Recommended Practices E 115 and E 116 were accepted by the Standards Committee on Nov. 9, 1959, and are available as separate reprints. The revision of tentative Definitions E 135 is still before the Standards Committee.[1]

SUGGESTED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the following suggested practices and methods:[2]

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The revision of Definitions E 135 was accepted by the Standards Committee on Sept. 16, 1960, and will appear in the 1960 Book of Methods for Emission Spectrochemical Analysis.

Suggested Recommended Practices for:

Installation and Safe Operation of the Spectrochemical Laboratory (Revised) (SM 1-1),
The Use of Statistical Methods in Spectrochemical Methods (SM 2-4),
Flame Photometric Methods of Spectrochemical Analysis (SM 2-5), and
Establishing and Controlling Spectrochemical Analytical Curves (SM 2-6).

Suggested Method for Spectrochemical Analysis of:

Beryllium Copper Alloys by the Rotating Disk Spark Technique (SM 5-11),
Zinc and Its Alloys by the Point-to-Plane Spark and Condensed Arc Techniques Using a Recording Photoelectric Spectrometer (SM 6-18),
Lead and Its Alloys by the Point-to-Plane Technique Using a Recording Photoelectric Spectrometer (SM 6-19),
Cast Iron by the Point-to-Plane Spark Technique (SM 9-12),
Plain-Carbon and Low-Alloy Steel by the Point-to-Plane Method (SM 9-13),
Plain-Carbon and Low-Alloy Steel by the Rod-to-Rod Spark Technique (SM 9-14),
Stainless Type Steels by the Point-to-Plane Spark Technique (SM 9-15),
Steel by the Solution - Rotating Disk - Spark Technique (SM 9-16),
Cast Iron for Cerium by the Point-to-Plane Condensed Arc Method (Sm 9-17),
Plain-Carbon and Low-Alloy Steel by the Pellet Technique (SM 9-18),
Cement for Manganic, Sodium, and Potassium Oxides by the Flame Photometer Technique (SM 10-16), and
Glass for Alkaline Earth Elements by the Flame Photometer Technique (SM 10-17).

NEW TENTATIVE

The committee recommends for publication as tentative the Recommended Practices for Spectrochemical Computations, as published in "Methods for Emission Spectrochemical Analysis (1957)," p. 72, with the following revisions:

Section 1.—In line 1 change "suggested" to "recommended."

Section 4(b).—Change the fourth sentence, beginning "However, in many optical . . ." to read "However, in many optical emission sources such as electrical arcs and sparks, the absolute intensities are variable, and can be used only when obtained by averaging a large number of runs."

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Recommended Practices for:

Designation of Shapes and Sizes of Preformed Graphite Electrodes (**E 130 – 57 T**).[3]

Tentative Methods for Spectrochemical Analysis of:

Nickel Alloys by the Powder - D-C Arc Technique (**E 129 – 57 T**),[3] and
Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (**E 101 – 53 T**).[4]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (A. W. Helz, chairman) has revised the suggested method for Installation and Safe Operation of the Spectrochemical Laboratory as referred to earlier in this report. It is (1) preparing tentative practices on describing and specifying the excitation source, and a suggested practice for describing and specifying the spectrograph; (2) compiling a summary of the characteristics of the various emulsions used throughout the world; (3) testing cut film of SA No. 1 emulsion; and (4) studying the possibility of writing a practice on the important characteristics of an X-ray spectrograph.

---

[2] These suggested methods and recommended practices will appear in the 1960 Book of Methods for Emission Spectrochemical Analysis.

[3] Issued as a separate reprint.

[4] 1956 Book of ASTM Methods for Chemical Analysis of Metals.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Fundamental Practices (R. W. Smith, chairman) has revised the two tentatives on photographic processing and photometry; recommended the acceptance of a tentative practice for spectrochemical computations; and submitted three suggested practices on statistics, analytical curve control, and flame photometry as reported earlier in this report. In addition, it is (1) assembling a bibliography in flame photometry; (2) preparing a recommended basic practice in X-ray spectroscopy; (3) assembling a bibliography in X-ray spectroscopy; (4) planning a cooperative test for X-ray analysis of steel; and (5) compiling a list of unsolved problems in spectrochemical analysis.

Subcommittee III, Editorial (D. C. Spindler, chairman) has (1) reviewed 50 terms and recommended revision of Definitions E 135 as reported earlier in this report; (2) has continuously considered possible changes in the E-2 Regulations and Recommendations to stay in line with current ASTM practice and the needs of the committee; (3) has reviewed 47 drafts of 34 methods, practices and reports; and (4) has assisted in final publication plans for the 16 suggested methods and practices, the revision of 6 tentatives, the annual report of the committee, and the proposed new edition (1960) of "Methods for Emission Spectrochemical Analysis" mentioned earlier in this report.

Subcommittee IV on Electrodes, Pure Materials, Reagents, and Standards (W. J. Edgar, chairman) is maintaining a current list of all standards and pure materials available throughout the world and is trying to determine the characteristics of graphite electrodes which are important to spectrochemical analysis.

A revision of the "Report on Standard Samples and Related Materials for Spectrochemical Analysis" is complete; publication is expected early in 1961.

Subcommittee V on Copper, Nickel and Their Alloys (A. W. Young, chairman) is (1) assisting the National Bureau of Standards in preparing copper alloy standards; (2) has prepared the suggested method SM 5-11 as reported earlier; (3) is preparing a suggested method for X-ray fluorescence analysis of copper-base alloys; and (4) is processing two other suggested methods.

Subcommittee VI on Lead, Tin, Zinc and Related Materials (J. F. Murphy, chairman) is (1) setting up cooperative testing methods for a suggested method on tin (SM 6-9) and a suggested method on zinc (SM 6-15); (2) investigating the possibility of solution methods for cadmium, bismuth, indium, and gallium; and (3) preparing two direct reading methods of analysis. It has submitted two new methods (SM 6-18, SM 6-19), as mentioned earlier in this report.

Subcommittee VII on Aluminum and Magnesium and Their Alloys (F. R. Potter, chairman) is (1) cooperatively testing SM 7-10 (Aluminum and Its Alloys by the Point-to-Plane Spark and Intermittent Arc Techniques, Using a Recording Photoelectric Spectrometer) and a new proposed tentative method for the spectrochemical analysis of magnesium alloys for calcium by the flame photometer technique; (2) revising an X-ray fluorescence analysis method; (3) determining the requirements for trace analysis with a direct reader; and (4) preparing two other suggested methods.

Subcommittee VIII on Titanium, Zirconium, and Related Alloys (N. E. Gordon, chairman) is (1) preparing two suggested methods on zirconium alloys and three suggested methods on titanium alloys; (2) considering the advisability of setting up task groups on columbium and molybdenum; and (3) working cooperatively with the Materials Branch of the Atomic Energy Commission and the National Bureau of Standards to obtain titanium alloy and zirconium alloy standards.

Subcommittee IX on Ferrous Alloys (J. F. Woodruff, chairman) has (1) submitted seven suggested methods for publication (SM 9-12, SM 9-13, SM 9-14, SM 9-15, SM 9-16, SM 9-17, and SM 9-18) as mentioned earlier in this report; (2) is cooperatively testing five methods in order to raise them to tentative; (3) is processing two spectrographic methods, one X-ray fluorescence method, and three direct reader methods; and (4) is preparing a suggested method for sampling ferrous materials.

Subcommittee X on Glass, Ceramics, Alkalies, and Cementitious Materials (W. M. Hazel, chairman) has prepared two suggested methods (SM 10-16, SM 10-17) as mentioned earlier in this report, and is working on a direct reader method for the analysis of alumina powder. It is also trying to obtain closer liaison with the C and D Committees and to obtain X-ray fluorescence methods.

Subcommittee XI on Slags, Ores, and Other Miscellaneous Nonmetallic Materials (S. R. Wiley, chairman) is preparing five suggested methods on the following materials: alumina-base materials, blast-furnace and open-hearth sinter, cerium minerals, general method for powdered samples, and blast furnace slags.

This report has been submitted to letter ballot of the committee, which consists of 204 members; 99 members returning their ballots, of whom 96 have noted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

DAVID L. FRY,
Chairman.

CYRUS FELDMAN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Recommended Practice for:

Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis (E 172 - 60 T).

Revision of Tentative Definitions of Terms Relating to:

Emission Spectroscopy (E 135 - 60 T).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the new and revised tentatives appear in the 1960 Book of ASTM Methods for Chemical Analysis of Metals.

REPORT OF COMMITTEE E-3
ON
CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held one meeting during the year: on June 24, 1959 in Atlantic City, N. J. in conjunction with the Annual Meeting of the Society. The Advisory Committee, divisions, and task groups held individual sessions June 22–24, 1959.

A Symposium on Extension of Sensitivity is planned jointly with Committee E-2 on Emission Spectroscopy for 1961.

New task groups on beryllium, molybdenum, and hafnium have been organized under Division M on Miscellaneous Metals.

Division S on Sampling has been requested to work on methods for lithium, and it is expected that in the near future the committee will concern itself with all the alkali metals.

The officers elected for the ensuing term of two years are as follows:

Chairman, Arba Thomas.
Vice-Chairman, J. L. Hague.
Vice-Chairman, R. G. Ernst.
Secretary, H. Kirtchik.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Proposed Tentative Methods for:

Chemical Analysis of Ferroboron, and
Chemical Analysis of Electronic Nickel.

Proposed Tentative Method of Test for:

Hydrogen Loss of Copper Powder.

REVISIONS OF TENTATIVES

The committee recommends revisions as appended hereto[1] of the following tentatives and their continuation as tentative:

Revision of Tentative Methods for:

Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (**E 30 – 59 T**),[2] and

Chemical Analysis of Titanium and Titanium-Base Alloys (**E 120 – 56 T**),[3] by the addition of new procedures for silicon, magnesium and tungsten, and the revision of the present Sections 6 to 12 on aluminum, the remainder of the method being adopted as standard as indicated later in the report.

Revision of Tentative Recommended Practice for:

Photometric Methods for Chemical Analysis of Metals (**E 60 – 50 T**).[3]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that Sections 1 to 5 and 13 to 92 of the Tentative Methods for Chemical Analysis of Titanium and Titanium-Base Alloys (**E 120 – 56 T**),[3] be approved for reference to letter ballot of the Society for adoption as standard with the following revisions:

Section 40(a)(3).—Change "10 min" in last sentence to "1 hr." Add at the end "In the case of vanadium alloys, allow to stand overnight at room temperature or place in a water bath at 60 to 70 C for 30 min, remove, and cool to room temperature."

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new and revised tentatives will appear in the 1960 Book of ASTM Methods on Chemical Analysis of Metals.

[2] Available as a separate reprint.

[3] 1956 Book of ASTM Methods for Chemical Analysis of Metals.

Section 69(b).—Change to read as follows:

(*b*) *Standard Molybdenum Solution* (*1 ml = 0.1 mg Mo*).—Dissolve 0.372 g of ammonium molybdate ($(NH_4)_6Mo_7O_{24} \cdot 4H_2O$) in 1800 ml of water. Transfer to a 2-liter volumetric flask, fill to the mark, and mix. Standardize as described for Reagent No. 8.

Section 72(c).—Change to read as follows:

(*c*) *Reference Solution.*—Use a freshly prepared reference solution made by adding 25 ml of $HClO_4$ to 25 ml of water (cool the solution after the addition of the $HClO_4$), followed by 30 ml of reagent mixture, prepared as directed in Section 69 (*h*), and dilute with water to a volume of 100 ml.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods for Chemical Analysis of:

Magnesium and Magnesium-Base Alloys (**E 35 – 58 T**),[2]
White Metal Bearing Alloys (**E 57 – 57 T**),[2]
Electronic Nickel (**E 107 – 56 T**),[3]
Copper-Chromium Alloys (**E 118 – 56 T**),[3] and
Copper-Tellurium Alloys (**E 121 – 56 T**).[3]

Tentative Method for Determination of:

Arsenic in Fire-Refined Copper (**E 62 – 58 T**).[2]

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as appended hereto[4] of the Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals (**E 50 – 53**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Methods of Chemical Analysis of:

Brasses (**E 36 – 45**),[3]
Copper (Electrolytic Determination of Copper) (**E 53 – 48**),[3] and
Silver Solders (**E 56 – 45**).[3]

Standard Methods for Sampling:

Ferro-Alloys for Determination of Chemical Composition (**E 32 – 42**).[3]

Standard Method of Sampling:

Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (**E 55 – 48**).[3]

The recommendations in this report have been referred to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF DIVISIONS

Division S on Sampling (A. C. Holler, chairman).—In the Ferrous Subcommittee, methods for the sampling of Ferro-Alloys (E 32) were editorially revised. A proposed addition to E 59 – 57 to cover the sampling of nodular cast iron has been submitted to Committee A-3 for review. A method for the sampling of gray iron so as to obtain a representative sample for the carbon determination is being studied in cooperation with Committee A-3. In the Non-Ferrous Subcommittee, work was initiated on methods of sampling of beryllium and its alloys. In the Miscellaneous Metals Subcommittee, work continues on sampling methods for lithium, molybdenum, titanium, and tungsten in cooperation with the B-Committees.

---

[4] The revised standard will appear in the 1960 Book of ASTM Methods for Chemical Analysis of Metals.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Division M on Miscellaneous Metals (G. Porter, chairman):

Columbium Metal

The task force review on methods for the determination of molybdenum, tungsten, tantalum, hydrogen, and nitrogen in columbium is completed and in good agreement with two exceptions which are being resolved. They should be ready for committee letter ballot by July. A method for oxygen is ready for testing but lack of suitable samples has delayed task force review. Testing of the method for titanium is practically complete. Suitable metal has been obtained and distributed for testing the methods for iron, zirconium, and boron. Further work and suitable samples are needed for the carbon method review. Samples have been obtained for the silicon tests but no suitable method is available.

Metal Powders

The method for Hydrogen Loss of Copper Powder referred to above was prepared by the task group. The review of the method for Hydrogen Loss of Iron Powder is complete. The small changes necessary to adapt methods now in the book to the chemical analysis of metal powders are being reviewed.

Molybdenum

The task force review on the determination of oxygen is complete and in good agreement. Unless the group feels the study should be extended to include tungsten, the work of this task force will be completed upon submission of the method.

Nickel-Chromium Alloys

A task force to revise the method for nickel now in the book has been formed. Methods for the other elements will be reviewed.

Titanium

Methods for magnesium, silicon (2) and tungsten being added to E 120 - 56 T were prepared by this group. Testing of oxygen (2), chloride, columbium, tantalum and carbon methods is complete. These methods were developed and tested by the Panel on Methods of Analysis of the Metallurgical Advisory Committee on Titanium.

Evaluation of the sodium hydroxide-oxine and double fluoride methods for aluminum is continuing and a new titration method has been distributed.

Zirconium

Methods for silicon, tin, and titanium will be available by July. The task force review on uranium, tungsten, carbon, and another tin method is practically completed. Further testing of the oxygen and boron methods is under way. Procedures for thorium, columbium, tantalum, and phosphorus are being reviewed prior to testing.

Division N on Non-Ferrous Metals (R. G. Ernst, chairman).—The following methods have been substantially completed and will be ready for inclusion in the new edition of the methods book: (1) Methods for the Analysis of Silver Brazing Alloys—determination of silver, copper, cadmium and zinc, (2) Method for the Determination of Boron in Aluminum-Base Alloys (Photometric), (3) Method for the Determination of Zinc in Magnesium-Base Alloys (by EDTA Titration), (4) Method for the Determination of Tin in Copper-Base Alloys (by Hypophosphite Reduction, without copper removal), and (5) Method for the Determination of Antimony in Copper-Base Alloys (Photometric).

In addition, methods for tungsten and magnesium in electronic nickel are being developed in cooperation with Subcommittee IX of Committee F-1.

Work is continuing on the editorial revision and modernization of the existing procedures for analysis of non-ferrous metals, taking advantage of the latest developments in the field of analytical chemistry. It is believed that considerable consolidation of methods can be made without any sacrifice of the utility of the book. A review of specifications is being made in order to determine where new or revised analytical methods are required to test for compliance.

A new task force has been organized on the analysis of beryllium metal and oxide.

Division F on Ferrous Metals (J. J. Furey, chairman) prepared the proposed Tentative Methods for Chemical Analysis of Ferroboron, and the revision of Tentative Methods E 30 referred to above.

Low-Carbon (less than 0.01 per cent) in Steel

Sample materials with carbon in the triple-O range have been obtained. Homogeneity testing is under way, and cooperative work will begin when these tests are completed. Provision for either induction or resistance heating will be made. It is hoped to have a tentative method ready for the 1960 edition of the book.

Manganese in Ferromanganese and Silicomanganese (Pyrophosphate Method)

Some interference was noted from the presence of about 1.0 per cent chromium in one of the samples. It was first thought that chromium should be volatilized before determining the manganese, but later it was found that a pH adjustment to 4.5 before titration would prevent chromium interference. Results are being evaluated and it is planned to have the method ready for ballot.

Ferrocolumbium

Preliminary tests indicate that an ion-exchange method developed by the National Bureau of Standards for the estimation of columbium, tantalum, and titanium in this alloy will give satisfactory results. The method is being written and will be distributed to the task force members together with a sample of the alloy. It is intended that an NBS sample of this alloy will be issued.

High Cobalt in Alloys

A method involving a caustic-bromine separation followed by titration with ferricyanide was tested in six laboratories. The spread in results was slightly too wide for acceptance.

Work is now under way to try to establish a primary cobalt standard, and a sample of cobalt metal has been distributed to check agreement between laboratories on the primary standard.

Aluminum in Steel (Photometric)

A method using eriochrome-cyanine-R has been tested, and data are being tabulated. A method involving use of 8-hydroxyquinoline is also under consideration.

Nitrogen in Steel (Soluble and Insoluble)

A method and samples have been submitted to the task force. Some difficulty has been reported with the photometric procedure.

Sulfur in Steel

A method to apply at levels below 0.01 per cent with an accuracy of 0.0001 to 0.0002 per cent is being sought. As to the type of method required, while it is not always possible to develop an absolute method, a method that will repeat well from laboratory to laboratory should be the objective. A method employing distillation-titration is being considered as the first step, with the final objective a combustion method similar to one now described.

Hydrogen in Steel

Results on charged steel samples and on charged nickel samples were not within acceptable limits. The work will continue, and a different approach will be considered. This assignment is a particularly difficult one and will require a great deal of hard work before the desired result is achieved.

The present methods for analysis of steel and ferro-alloys were reviewed, editorial corrections made where necessary with special attention to the use of a common practice where possible.

Division G on General Analytical Methods (L. M. Melnick, chairman).—A review has been made of many of the existing methods. Revisions of E 50 and E 60 are appended to the report. Planning is now under way for a Symposium on Extension of Sensitivity in 1961, jointly with Committee E-2. Four speakers have been obtained.

Editorial Subcommittee (J. L. Hague, chairman).—Two meetings were held during the year. A new policy is being considered for methods emanating from other technical committees. It was deemed advisable that the chairman of Committee E-3 be first made cognizant of such methods. These could be officially received and then delegated to the proper division for further action. Liaison at the divisional level would follow.

This report has been submitted to letter ballot of the committee, which consists of 117 members; 59 members returned their ballots, of whom 58 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ARBA THOMAS,
Chairman.

H. KIRTCHIK,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods for Chemical Analysis of:
Aluminum and Aluminum-Base Alloys (E 34 – 60 T), and
Special Brasses and Bronzes (E 54 – 60 T).

New Tentative Method for Test of:
Antimony in Copper and Copper-Base Alloys (E 62 – 60 T), and
Oxygen in Molybdenum (E 174 – 60 T).

New Tentative Recommended Practices for:
Conducting Inter-Laboratory Studies of Methods for Chemical Analysis of Metals (E 173 – 60 T).

Revision of Tentative Methods for Chemical Analysis of:
Ferroboron (E 31 – 60 T),
Titanium and Titanium-Base Alloys (E 120 – 60 T), and
Zirconium and Zirconium-Base Alloys (E 146 – 59 T).

Revision and Reversion to Tentative of Standards Methods for Chemical Analysis of:
Magnesium and Magnesium-Base Alloys (E 35 – 60), and
Silver Solders (E 56 – 45).

These recommendations were accepted by the Standards Committee on December 29, 1960, and the new and revised tentatives appear in the 1960 Book of ASTM Methods for Chemical Analysis of Metals.

REPORT OF COMMITTEE E-4 ON METALLOGRAPHY*

Committee E-4 on Metallography, together with several of its subcommittees, held two meetings during the past year: in Atlantic City, N. J., on June 21 to 24, 1959, and in Chicago, Ill., on February 1 to 3, 1960.

The committee consists of 117 members.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. L. Wyman.
Vice-Chairman, R. E. Penrod.
Secretary, Mary R. Norton.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

- Preparation of Micrographs of Metals and Alloys (Including Recommended Practices for Photography as Applied to Metallography) (**E 2 - 49 T**),
- Preparation of Metallographic Specimens (**E 3 - 58 T**),
- Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (**E 79 - 49 T**),
- Preparing Quantitative Pole Figures of Metals (**E 81 - 54 T**),
- Determining the Orientation of Metal Crystal (**E 82 - 49 T**),
- Estimating the Average Grain Size of Non-Ferrous Metals, Other than Copper, and Their Alloys (**E 91 - 51 T**).

Tentative Definitions of:

- Terms Relating to Metallography (**E 7 - 55 T**).

Tentative Recommended Practice for:

- Thermal Analysis of Metals and Alloys (**E 14 - 51 T**),
- Identification of Crystalline Materials by the Hanawalt X-ray Diffraction Method (**E 43 - 49 T**), and
- Dilatometric Analysis of Metallic Materials (**E 80 - 49 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (W. D. Forgeng, chairman) is currently sponsoring a symposium on the polishing of metallographic samples,[1] with a view to incorporating the latest and best practices available into further modifications of Methods E 3.

Subcommittee III on Nomenclature (W. L. Fink, chairman) is surveying the comments received and present opinions concerning the proposed system of nomenclature for phase designations in metallic systems. The consensus thus far of metallurgists in this country continues to be highly favorable; the survey will be continued through personal contacts with European metallurgists during this next summer.

Subcommittee IV on Photography (G. A. Ellinger, chairman) has reviewed Methods E 2 in detail and has decided that no revision is necessary at this time.

Subcommittee VI on X-ray Methods (R. K. Scott, chairman) has received the draft of the new version of Recommended Practice E 43 which was prepared by Messrs. Christ, Matthews and Parrish. The new text is extremely comprehensive in its coverage of the several phases of powder diffraction work and is too voluminous for inclusion in the Book of Standards. The draft is now being edited, with the intent that it will be published as a special technical publication. The Book of Standards will contain only a brief outline of its revised contents.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Symposium on Methods of Metallographic Specimen Preparation, *ASTM STP No. 285*.

In connection with the cooperative effort in joint committee work, it may be reported that editorial work has been completed on the tenth set of cards for the X-ray file, and work is progressing on the books and cards for the new editions of sets 1 to 5.

Subcommittee VII on Thermal Analysis (D. I. Finch, chairman) has drafted a recommended practice for resistometric (emf) analysis of metallic materials which is now being balloted by the subcommittee.

Subcommittee VIII on Grain Size (R. E. Penrod, chairman) has recommended the inclusion of additional data in Methods E 112 that will make the information contained in the tables much more usable. The subcommittee has been actively engaged in the preparation of micrographs for Plate II of these Methods.[2]

Subcommittee IX on Inclusions (Samuel Epstein, chairman) has been actively engaged in a detailed study of the changes and additions necessary to Recommended Practice E 45 so that it can serve adequately to evaluate the inclusion contents of modern alloys. Some of these changes are being incorporated into E 45 this year, which will occasion its reversion from standard to tentative. Further additions and necessary amplifications of the comparison chart are planned before the next publication of the Book of Standards.

Subcommittee XI on Electron Microstructure of Metals (N. A. Nielsen, chairman).—Task Group C has completed the progress report on "Electron Microstructure of Precipitation Hardenable Austenitic and Nickel-Base Alloys," which summarizes the electron metallographic work carried out by several subcommittee members on Inconel X and W-545 alloys.[3] The report, first presented at the open technical session which Subcommittee XI sponsored at the 1959 Annual Meeting, has more recently been presented before the annual meeting of the German Society for Electron Microscopy held in Freiberg, Germany, Oct. 18 to 21, 1959. This was the second such instance of a report of Subcommittee XI being presented at a European meeting on electron microscopy.

The open technical session consisted of twelve research papers dealing with electron microscopy, electron diffraction, and electron probe microanalysis. These papers were deemed of sufficient interest and merit to justify their publication by ASTM as a bound volume in the Society's STP series. The publication became available for purchase in March, 1960, under the title "Symposium on Electron Metallography" (*ASTM STP No. 262*).

A technical session on electron metallography is being sponsored by the subcommittee at the 1960 Annual Meeting.

Several of the task group activities have been consolidated to concentrate efforts on techniques and studies pertaining to the examination of aluminum, iron, stainless steel, and other alloys by transmission techniques.

Task Group D on the electron micro-probe analysis has held several meetings and is currently focusing its efforts in two directions, one being the preparation and collection of a series of solid solution alloys to serve as calibration standards, the other being the round-robin studies of several diffusion couples, samples of which have been provided by committee members.

---

[2] It is expected that copies of this plate will be available late in 1960 or early in 1961.

[3] This progress report has been included in Symposium on Electron Metallography, Am. Soc. Testing Mats., p. 3 (1960). (Issued as separate publication *ASTM STP No. 262.*)

The adaptability of modern electron microscopes to electron diffraction techniques has intensified interest in this phase of metallographic investigation. As a consequence a new group primarily concerned with electron diffraction is being formed in Subcommittee XI during the current Annual Meeting.

Subcommittee XIV on Quantitative Metallography.—Due to the increasing demands for the quantitative evaluation of microstructures, coupled with improved techniques in this area, it was the decision of the E-4 Advisory Committee at the February meeting that a new subcommittee be formed to devote its attention to the subject.

This report has been submitted to letter ballot of the committee, which consists of 117 members; 84 members returned their ballots, of whom 81 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY R. NORTON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Methods for:

Estimating the Average Grain Size of Metals (E 112 - 58 T).

Revision and Reversion to Tentative of Recommended Practice for:

Determining the Inclusion Content of Steel (E 45 - 51).

These recommendations were accepted by the Standards Committee on September 29, 1960, and the revised tentative methods and recommended practice appear in the 1960 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE E-5
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION*

Committee E-5 on Fire Tests of Materials and Construction held one meeting during the year: in Chicago, Ill., on Feb. 3, 1960, during ASTM Committee Week.

The committee consists of 69 members, of whom 32 are classified as producers, 7 as consumers, and 30 as general interest members, with 2 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. D. Foster.
Vice-Chairman, G. W. Shorter.
Secretary, I. A. Benjamin.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Method of Test for Measuring the Surface Flammability of Building Materials Using an 8-ft Tunnel Furnace, as appended hereto.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and definitions as appended hereto (see Editorial Note):

Tentative Methods of:

Fire Tests of Window Assemblies, and
Test for Measuring Surface Flammability of Materials Using a Radiant Heat Energy Source.

Tentative Definitions of:

Terms Relating to Fire Tests of Building Construction and Materials.

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Method of Test for Surface Burning Characteristics of Building Materials (**E 84 - 59 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard with revisions as appended hereto (see Editorial Note).

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions issued June 1959[2] of Sections 9 and 30 of the Standard Methods of Fire Tests of Building Construction and Materials (**E 119 - 58**)[3] be approved for reference to letter ballot of the Society for adoption as standard (see Editorial Note).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] See p. 462.

[2] 1959 Supplement to Book of ASTM Standards, Part 5.

[3] 1958 Book of ASTM Standards, Part 5.

[4] The recommendations in this report were not accepted at the Annual Meeting due to the absence of a quorum; see Editorial Note for later acceptance by the Administrative Committee on Standards.

This report has been submitted to letter ballot of the committee, which consists of 69 members; 61 members returned their ballots, of whom 56 have voted affirmatively and 3 negatively.

Respectfully submitted on behalf of the committee,

W. J. Krefeld,
Chairman.

H. D. Foster,
Secretary.

Editorial Note

At the Annual Meeting Session at which the Report of Committee E-5 was presented, objection was raised to one of the recommendations. The question of quorum was raised and since no quorum was present, no actions on the report could be taken. Subsequent to the Annual Meeting, Committee E-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of:

Test for Measuring the Surface Flammability of Materials Using a Radiant Heat Energy Source (E 162 - 60 T), and

Fire Tests of Window Assemblies (E 163 - 60 T).

New Tentative Definitions of Terms Relating to:

Fire Tests of Building Construction and Materials.

Revision of Tentative Method of Test for:

Surface Burning Characteristics of Building Materials (E 84 - 59 T).

The new Tentative Methods E 162 and E 163, and the revision of Tentative Method E 84 were accepted by the Standards Committee on September 29, 1960, and appear in the 1960 Supplement to Book of ASTM Standards, Part 5. The proposed tentative definitions were referred back to Committee E-5 for further consideration in light of negative votes which had been cast in the committee.

APPENDIX

PROPOSED METHOD OF TEST FOR SURFACE FLAMMABILITY OF BUILDING MATERIALS USING AN 8-FT TUNNEL FURNACE[1, 2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test covers a procedure for measuring and comparing surface flame spread of various building materials, including protective and decorative coatings applied to these materials. The amount of smoke and heat produced in burning under this standard test condition is also measured. The results of the method may be used as an index to the fire hazard of such materials. The method is applicable to all building materials and composites that are capable of being mounted and supported within a 14-in. by 8-ft test frame so as to provide exposed surfaces over which flames may spread.

Apparatus

2. (*a*) The apparatus shall consist essentially of a gas-heated tunnel furnace about $10\frac{1}{2}$ ft long with a separate combustion chamber to accommodate a test specimen $13\frac{3}{4}$ in. wide by 8 ft long. General views of the exterior of the tunnel furnace and of the specimen combustion chamber are given in Figs. 1 and 2.[3]

(*b*) The furnace shall slope at a 6-deg angle from end to end and have three compartments: the firebox (item *2*, Fig. 1) extending the entire length of the furnace; the specimen combustion chamber (above the partition plate, item *5*, Fig. 2); and the hood and stack (items *6* and *7*, Fig. 1).

(*c*) A 12-gage, type 310 stainless steel partition (item *5*, Fig. 2) shall be located between the firebox and the combustion chamber. Thirty-three holes, $1\frac{19}{32}$ in. in diameter and $2\frac{9}{16}$ in. apart center to center, shall be located as shown in this partition. Meker burner tops shall be set into each of these holes. Asbestos paper washers $\frac{1}{32}$ in. thick shall be inserted within each of the Meker burner tops Nos. 14 through 33 to graduate the openings as follows:

Hole No.	Size, in.	Hole No.	Size, in.
1 to 13	23/16	24, 25	10/16
14, 15	16/16	26, 27	9/16
16, 17	14/16	28, 29	8/16
18, 19	13/16	30, 31	7/16
20, 21	12/16	32, 33	6/16
22, 23	11/16		

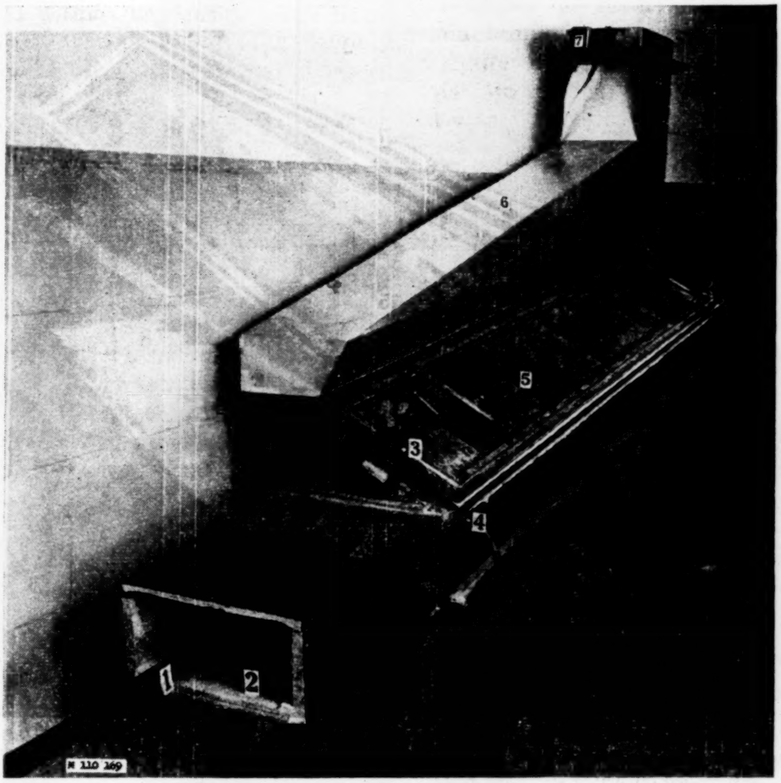
---

[1] This proposed method is under the jurisdiction of the ASTM Committee E-5 on Fire Tests of Materials and Construction.

[2] Published as information, June, 1960.

[3] Detail drawings of this apparatus are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

(*d*) The body of the furnace shall be constructed of 12-gage, low-carbon steel lined with high-grade asbestos millboard 1 in. thick. The hood and stack (item *6*, Fig. 1) shall be of 16-gage, low-carbon steel lined with asbestos millboard $\frac{1}{4}$ in. thick. The stack of the furnace shall be located beneath a ventilating hood with sufficient draft to remove accumulating smoke. There should be no direct connection between the stack and ventilating hood since the only draft within the furnace shall be that developed from the burning of the heating gas and test specimen.

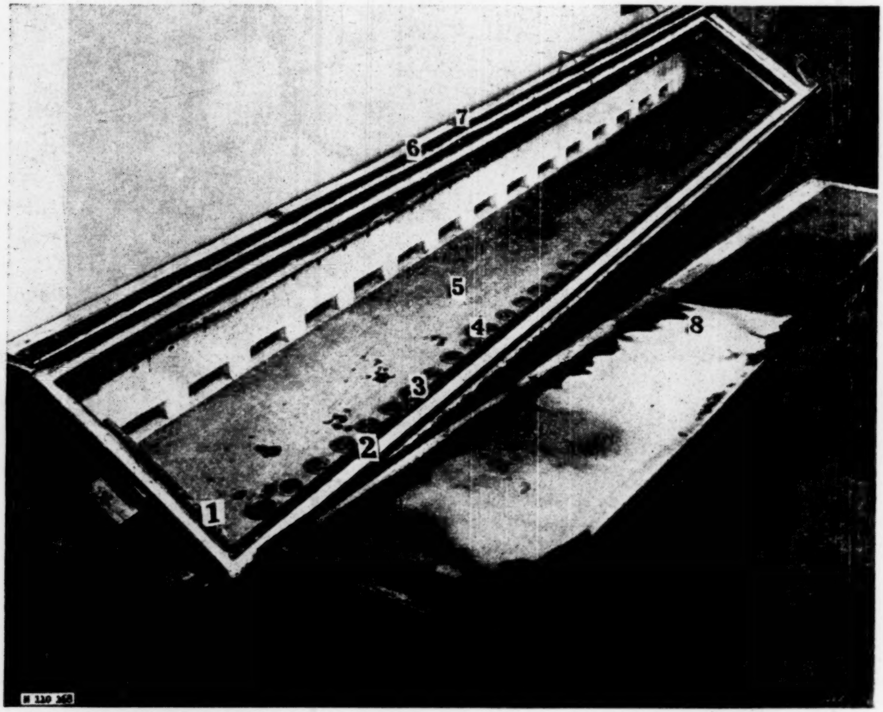


1—Gas supply to main burner.
2—Firebox.
3—Clamp to hold down cover over test specimen.
4—Gas supply to igniting burner.
5—Cover over test specimen.
6—Hood to collect combustion gases for temperature and smoke measurement.
7—Photoelectric cell for smoke density measurement.

FIG. 1.—Specimen Side of FPL 8-ft Tunnel Furnace.

(*e*) The temperature and smoke density of the combustion gases shall be measured in the stack. For the temperature measurement, two thermocouple junctions shall be embedded in a copper rod, $\frac{1}{2}$ in. in diameter and $17\frac{1}{8}$ in. in length, held horizontally in the stack (item 7, Fig. 1). Temperatures of the thermocouple junctions shall be measured by a potentiometer. Smoke density shall be determined with a photoelectric type of smoke meter, which indicates the reduction in the intensity of a column of light passing horizontally through the stack (item 7, Fig. 1) to a photoelectric cell.

(*f*) The main burner (item *1*, Fig. 1) shall consist of a $1\frac{1}{4}$-in. T-head iron pipe in which are drilled two parallel rows of holes 90 deg apart. Each row shall have 53 holes, $\frac{1}{8}$ in. in diameter with centers $\frac{1}{4}$ in. apart (Note 1). The burner shall be located in the firebox, with the holes 13 in. from the front end and the top of the pipe $2\frac{1}{8}$ in. below the bottom surface of the steel partition. The igniting burner shall consist of a $\frac{1}{4}$-in. iron pipe in which have been drilled six holes $\frac{1}{8}$ in. in diameter with centers 1 in. apart. The igniting burner shall be located $\frac{1}{2}$ in. below and parallel to the face of the test panel and 1 in. from the lower end of the panel.



1—**Igniting burner.**
2—Sand trough to seal cover.
3—Angle-iron bed on which specimen rests.
4—Holes in hot plate inset with Meker burner tops.
5—Hot plate over firebox.
6—Sand trough to seal edge of hood.
7—Slot for escape of combustion gases.
8—Specimen cover.

FIG. 2.—Specimen Combustion Chamber of FPL 8-ft Tunnel Furnace.

NOTE 1.—This burner is suitable for natural gas. For bottled and manufactured gases, the burner construction may have to be altered.

Test Specimens

3. The test specimens shall be $13\frac{3}{4}$ in. wide by 8 ft long and conditioned to constant weight at a temperature of 80 ± 10 F and a relative humidity of 30 ± 5 per cent.

Comparison Standards

4. (*a*) The red oak comparison standard, to which the index value of 100 is arbitrarily assigned, shall consist of plain-sawed, select-grade red oak flooring selected within the density range from 37.0 to 41.0 lb per cu ft. The flooring shall be nailed to a backing of plywood $\frac{1}{4}$ in. thick.

(*b*) The asbestos millboard comparison standard, to which the index value of 0 is arbitrarily assigned, shall be $\frac{1}{4}$ in. thick and of sufficiently high quality to withstand the test conditions without sagging.

Procedure

5. (*a*) Before installing a test specimen, measure the temperature of the top surface of the steel partition (item *5*, Fig. 2) at a point midway between the sides of the partition and the ends of the specimen opening. Measure the temperature by means of a thermocouple junction placed at the designated point and covered by an asbestos pad 0.4 in. thick and 6 in. square. If, after a period of at least 2 min, the temperature is not 85 ± 5 F, a preheating flame (such as from the main burner) or a cooling draft (such as from a fan) may be used in the firebox to obtain the desired temperature. If a preheating flame or cooling draft is used, it should be stopped before the thermocouple and pad are positioned.

(*b*) When the temperature of the partition is within the prescribed limits, lay the test specimen on the angle-iron frame (item *3*, Fig. 2) tilted 30 deg from the horizontal. Then lay the specimen cover (item *5*, Fig. 1) over the specimen in the same frame and clamp it in place.

(*c*) Adjust the gas to the main burner (item *1*, Fig. 1) to a rate computed to give 3400 Btu per min with a supply of primary air adequate to produce a blue flame, introduced by an atmospheric injector air-gas mixing unit.

(*d*) Obtain from the gas supplier the current heat value of the gas in British thermal units per cubic foot under selected conditions of temperature and pressure. Measure the temperature and pressure of the gas as it is fed into the main burner and use these values to compute the volume rate of flow for 3400 Btu per min in the firebox by the equation:

$$V = \frac{P_1 T_2 3400}{P_2 T_1 H}$$

where:

V = desired rate of gas flow in cubic feet per minute under conditions of use for 3400 Btu per min,

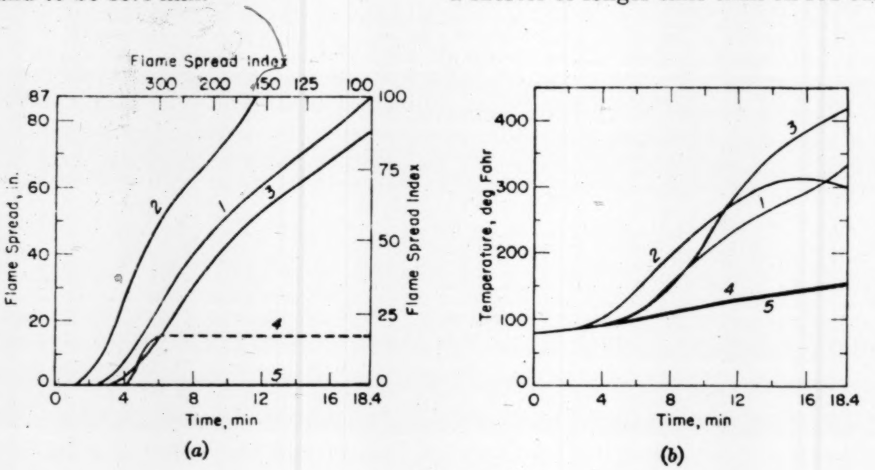
T_2 = absolute temperature of gas used, in degrees Kelvin (gas temperature in degrees Centigrade plus 273),

P_2 = absolute pressure of gas used, in millimeters of mercury (barometer pressure plus gage pressure), and

H = heat content of gas, in British thermal units per cubic foot under given conditions of P_1 millimeters of mercury and T_1 degrees Kelvin.

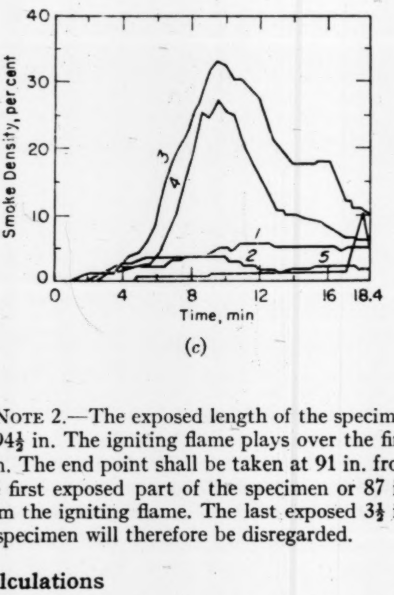
(*e*) Burn the gas in the igniting burner with no primary air at the rate of 85 Btu per min. The individual flames from the orifices in this burner play over the first 4 in. of an asbestos millboard specimen. Light both burners as simultaneously as possible, and start the stop watches at the instant of lighting.

(*f*) Watch the progress of the flame through the observation holes located along the side, with the line of sight towards the centerline of the specimen. As the flame front passes each observation hole, note the time and the distance traveled. Note temperatures and smoke densities every 30 sec. The observations should be plotted in graphs as shown in Fig. 3. The maximum length of travel of the flame on the specimen shall be 87 in. (Note 2). The average time for the flame to travel this distance on the red oak standard is the test period. It has been found to be 18.4 min.



(a)

(b)



(c)

FIG 3.—Typical Curves of Tunnel Data from Tests on (*1*) Red Oak, (*2*) Fiberboard A, (*3*) Hardboard M_1, (*4*) Gypsum Wallboard G, and (*5*) Asbestos Millboard.

NOTE 2.—The exposed length of the specimen is 94½ in. The igniting flame plays over the first 4 in. The end point shall be taken at 91 in. from the first exposed part of the specimen or 87 in. from the igniting flame. The last exposed 3½ in. of specimen will therefore be disregarded.

Calculations

6. (*a*) *Flame Spread Index.*—Express the flame spread as an index relative to the rate on red oak standard with an index of 100 and on asbestos board with an index of 0. Express the flame spread index, I_s, in either of two ways (Fig. 3(*a*)), depending on whether the flame has reached the end of the test specimen in a shorter or longer time than on red oak.

(*1*) For flame spread faster than on red oak:

$$I_s = \frac{T_o}{T_s} \times 100$$

where:

T_o = time to reach end of red oak specimen, and

T_s = time to reach end of test specimen.

(*2*) For flame spread slower than on red oak:

$$I_s = \frac{D_s}{D_o} \times 100$$

where:

D_s = distance reached on test specimen in test period, and

D_o = distance reached on red oak in test period.

(*b*) *Fuel Contributed Index.*—Obtain the fuel contributed index, I_c, by planimetering the areas under the curves indicated in Fig. 3(*b*) and calculating the index value as follows:

$$I_c = \frac{A_s - A_a}{A_o - A_a} \times 100$$

where:

A_s = area under specimen curve,

A_a = area under asbestos curve, and

A_o = area under red oak curve.

(*c*) *Smoke Density Index.*—Obtain the smoke density index, I_d, by planimetering the curves indicated in Fig. 3(*c*), and calculating the index value as follows:

$$I_d = \frac{A_s - A_a}{A_o - A_a} \times 100$$

Report

7. The report shall include the following:

(*1*) Identification of the test panel, including manufacturer and code designation, and, when known, the thickness, density, and composition of the panel,

(*2*) Identification, when known, of the composition, thickness or weight retained, of any chemicals added or coating applied to the test panel,

(*3*) Moisture content of the panel at start of test,

(*4*) Flame spread index, fuel contributed index, and smoke index as calculated in Section 6, including flame travel, temperature, and smoke density data taken during the test, and

(*5*) Record of observations made during the test as to appearance of flame, char pattern, and any intumescence or disintegration of panel or applied coatings.

REPORT OF COMMITTEE E-6
ON
METHODS OF TESTING BUILDING CONSTRUCTIONS*

Committee E-6 on Methods of Testing Building Constructions held two meetings during the year: in Chicago, Ill., during ASTM Committee Week, and in San Francisco, Calif., during the Pacific Area National Meeting.

A Symposium on Methods of Testing Building Constructions was held during the San Francisco meeting, and the papers presented will be published as *ASTM STP No. 282.*

The committee consists of 101 members, of whom 84 are voting members; 36 are classified as producers, 6 as consumers, and 42 as general interest members.

The establishment of a new Subcommittee on Curtain Wall Construction has been recommended.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. F. Legget.
Vice-Chairman, W. F. Aikman.
Secretary, J. P. Thompson.
Membership Secretary, R. W. Bletzacker.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Methods of Testing Materials for Use as Vapor Barriers Under Concrete Slabs and as Ground Cover in Crawl Spaces (**E 154 - 59 T**):[1]

Section 6.—Combine the center heading preceding this section with that preceding Section 8, to read as follows: "Resistance to Transmission of Water Vapor After Alternate Wetting and Drying and to Deterioration from Long-Time Soaking."

Add a new Paragraph (*d*) to read: "(*b*) *Mandrel.*—A bar or rod 1 in. in diameter and 18 in. long."

Section 7.—Change to read as follows:

7. ***Procedure.***—(*a*) Cut three specimens, 12 by 12 in., of the material to be tested.

(*b*) Immerse the specimens in potable water kept at a temperature of 73.4 ± 3.5 F (23 ± 2 C) for 16 hr (overnight). Then dry the specimens in an oven kept at 140 ± 3.5 F (60 ± 2 C) for 8 hr. Repeat the wetting and drying cycle for a total of 5 cycles (Monday through Friday) to be followed by immersing the specimens in water over the week end (64 hr). Repeat the wetting and drying cycle 5 more days and immerse the specimens in water for a period of 16½ days (week end plus 2 weeks). Dry the specimens at 140 ± 3.5 F for 8 hr, then condition to a constant weight in the chamber where water-vapor transmission tests are made.

(*c*) Cut the specimens in half, parallel to the machine direction (the long direction as taken from the roll or package). Bend one of the halves of each specimen with one surface up and the other specimen with the opposite surface up at an angle of 90 deg over a 1-in. diameter mandrel in a period of 2 sec or less (at 73.4 F). Record evidence of cracking or delamination.

(*d*) Cut specimens for the water-vapor transmission test so that the full bent portion is installed in the center of the pan with the surface that was concave at the time of bending facing the water. Use the test method described in Section 5.

Sections 8 and 9.—Delete these sections, renumbering subsequent sections accordingly.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1959 Supplement to Book of ASTM Standards, Part 5.

Section 11(c).—Change to read as follows:

(*c*) Remove the specimens from water, one at a time, lightly blot the free water from both surfaces, and immediately determine the tensile strength and stretch at maximum load. Test both sets of specimens at a temperature of 23 ± 2 C (73.4 ± 3.5 F) with the tester adjusted so that the distance between the jaws is about $\frac{1}{2}$ in. The jaws of the clamp should be lined with emery cloth or other material to prevent slipping of the specimen during test. Materials with very little stretch may be tested with a requirement for tensile strength only. Materials with high stretch may be tested with a requirement for energy absorbed to maximum load, in inch-pounds or other convenient unit. Average the 10 readings, crosswise and lengthwise, respectively.

Section 13(b).—Change to read as follows:

(*b*) *Standardization of Microbiological Activity of Soil.*—As a check on the microbiological activity of the soil, place in the test at the same time and in the same way as hereafter described for the membranes to be tested, cards of $\frac{1}{16}$-in. sweetgum-sapwood veneer that have been conditioned to a constant weight at the same temperature and relative humidity as maintained in the chamber where vapor transmission tests are made, as called for in Paragraph (*g*). Conditioning should take about 24 hr and the cards should have a moisture content of about 9 per cent. Individual cards should be removed from the test at intervals to establish a weight-loss trend. (Cards should be gently washed free of soil and conditioned before weighing.) When a veneer card has lost on the average no less than 35 per cent of its initial weight, the test is to be characterized as standard and thus the normal 6-week incubation period may be shortened or lengthened as necessary to accomplish this amount of weight loss. Control in this respect can be guided by using a small surplus of veneer cards and removing one from test at suitable intervals.

Section 15(a).—Add the following sentence: "Condition specimens at 73.4 ± 3.5 F (23 ± 2 C) and 50 per cent relative humidity to a constant weight."

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

Activities of Subcommittees

Subcommittee I on Segments of Wall, Floor, and Roof Constructions (J. A. Liska, chairman).—This subcommittee, formerly on Panels for Light Building Construction, has changed its scope to read as follows:

> To formulate, develop, and improve standard methods of test that will accurately measure the important structural properties of space-enclosing elements for building construction; provide a systematic basis for obtaining engineering data for these segments, and provide information for building officials, code authorities and others on which performance standards can be established.

The subcommittee expects to review Methods E 72 to clarify its presentation of the several test methods it contains.

Subcommittee II on Durability (E. C. Shuman, chairman) has recognized that standards to evaluate durability of building constructions would be practical only in the form of recommended practices. A British journal on this subject has come to the attention of the subcommittee and is being reviewed with interest.

Subcommittee III on Sound Transmission (R. K. Cook, chairman) is considering a revision of Recommended Practice E 90.

Subcommittee IV on Strength of Masonry Units (H. C. Plummer, chairman) is circulating a revised draft of a proposed test method for leakage through masonry panels. This subcommittee is also considered the sponsorship of a Symposium on Masonry Testing—possibly at the ASTM Annual Meeting in June 1961.

Subcommittee V on Trusses, Beams

---

[2] The letter ballot vote on this recommendation was favorable; the results of the ballot are on record at ASTM Headquarters.

and Columns (D. E. Kennedy, chairman).—This committee, formerly on Light Trusses, has changed its scope to read as follows:

> To formulate, develop, and improve standard methods of test that will accurately measure the important structural properties of trusses, beams, columns and other structural framing elements for building construction in order to provide information to building officials, code authorities and others on which performance standards can be established.

A review of Methods E 73 is in progress to make it more useful to engineers and code officials.

Subcommittee VI on Completed Structures (W. R. Schriever, chairman) has shortened its title from Load Tests on Completed Structures and changed its scope to read:

> To formulate, develop and improve standard methods of test that will measure the important structural properties of completed structures, in order to provide information for building officials, code authorities and others upon which performance standards can be established.

This subcommittee is developing a test method for loading tests on floors and flat roofs.

Subcommittee VII on Vapor Barriers Beneath Concrete Slabs on the Ground (L. W. Wood, chairman) developed the revisions in Methods E 154 listed earlier in this report. Other prospective revisions were recommended for further study.

Subcommittee VIII on Windows (L. M. Dunn, chairman).—The scope of this subcommittee was altered to cover operable windows only, but the area of interest was expanded to include window hardware. Seven task groups were established to consider structural performance, air infiltration, water leakage, thermal performance, hardware, definitions, and collection of existing performance requirements and methods.

This report has been submitted to letter ballot of the committee, which consists of 84 voting members; 65 returned their ballots, of whom 64 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. F. LEGGET,
Chairman.

R. A. BIGGS,
Secretary.

REPORT OF COMMITTEE E-7
ON
NONDESTRUCTIVE TESTING*

Committee E-7 on Nondestructive Testing met at Atlantic City, N. J., on June 25, 1959, in conjunction with the Annual Meeting of the Society. Seven subcommittees, the Executive Council, and several task groups also met at that time. Another series of meetings was held in Chicago, Ill., in February, 1960, during ASTM Committee Week.

Sidney Low has resigned as chairman of Subcommittee II on Reference Radiographs. The appointment of a new chairman is under consideration.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. H. Bly.
Vice-Chairman, C. H. Hastings.
Secretary, A. Gobus.

The Symposium on Nondestructive Testing in the Missile Industry sponsored by Committee E-7 for the ASTM Third Pacific Area National Meeting held in October, 1959, is being published by the Society as a Special Technical Publication, *ASTM STP No. 278.*

In connection with the Third International Conference on Nondestructive Testing held in March, 1960 at Tokyo, Japan, many of the E-7 sponsored technical publications and standards were exhibited by the ASTM with the cooperation of R. A. Pulk of the Detroit Arsenal. These publications were presented to the Japanese Society for Nondestructive Inspection at the end of the conference.

RECOMMENDATION ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On March 3, 1960, the Administrative Committee on Standards, acting for the Society, accepted the Tentative Reference Radiographs for Inspection of Aluminum and Magnesium Castings (Series II) (**E 155 - 60 T**). The introductory text for these radiographs will appear in the 1960 Book of ASTM Standards, Part 3. The radiographs will appear in a ring binder available from ASTM Headquarters at a price of $125.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods for:

Liquid Penetrant Inspection, and
Ultrasonic Contact Inspection of Weldments.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Reference Radiographs for:

Inspection of Aluminum and Magnesium Castings (**E 98 - 53 T**), and
Steel Welds (**E 99 - 55 T**).

Tentative Reference Photographs for:

Magnetic Particle Indications on Ferrous Castings (**E 125 - 56 T**).

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 3.

Tentative Methods for:

Dry Powder Magnetic Particle Inspection (**E 109 - 57 T**), and
Wet Magnetic Particle Inspection (**E 138 - 58 T**).

Tentative Recommended Practice for:

Ultrasonic Testing by the Resonance Method (**E 113 - 55 T**),
Ultrasonic Testing by the Reflection Method, Using Pulsed Longitudinal Waves Induced by Direct Contact (**E 114 - 55 T**), and
Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (**E 127 - 58 T**).

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Radiographic Recommended Practice (G. R. Forrer, chairman) is continuing its task of revising the Recommended Practice for Radiographic Testing (E 94 - 52 T). The investigation of changes in image appearance of radiographs exposed at varying distances from the same and different radiation sources is almost complete.

Subcommittee II on Reference Radiographs (new chairman being considered) was responsible for developing the Reference Radiographs for Inspection of Aluminum and Magnesium Castings (Series II) (E 155 - 60 T), mentioned earlier in this report. Several committees of the Aerospace Industries Assn. cooperated in this task.

The Task Group on Reference Radiographs for Heavy Steel Castings (F. H. Allison, Jr., chairman) is nearing completion of the radiographs for 3-in. sections. Also the preparation of radiographs for 6-in. and 12-in. sections is under way. In addition to the $2200 granted by the ASTM Administrative Committee on Research for this work, an additional $5000 has been donated by industrial firms and laboratories.

The Task Group on Brazed Honeycomb Construction (J. Fenton, chairman) is considering a preliminary draft covering terminology and reference radiographs for this type of construction.

The Task Group on Reference Radiographs for Bronze Castings (S. Goldspiel, chairman) has established a definite need for such reference radiographs. The present Bureau of Ships radiographic standards will serve as a basis for a committee sponsored document.

The Task Group on Reference Radiographs for Thin Wall Steel Castings (C. G. Poehlman, chairman), as a result of suggestions from the Naval Ordnance Laboratory and the Aerospace Industries Assn., is developing a draft document primarily for steel castings used by the aircraft industry. The scope of this group is being reconsidered with the thought of limiting it to precision investment steel castings.

The subcommittee has recommended the continuation as tentative of Reference Radiographs E 98 and E 99.

Subcommittee III on Magnetic Particle and Penetrant Testing (H. Migel, chairman) has developed the proposed Tentative Methods for Liquid Penetrant Inspection appended to this report. The subcommittee is collecting reference photographs for penetrant inspection similar to those for magnetic particle inspection published in the present Reference Photographs for Magnetic Particle Indications on Ferrous Castings (E 125 - 56 T). Any such photographs should be directed to V. I. Wiegand, 1943 Green Pine Drive, Cincinnati 31, Ohio.

The subcommittee has recommended the continuation as tentative of Methods E 109 and E 138 and Reference Photographs E 125.

Subcommittee V on Radiographic Procedure (E. L. Criscuolo, chairman) is cooperating with Subcommittee I on a program to consider the limitations and possible improvements of the penetrameter described in the Method for Controlling Quality of Radiographic Testing (E 142 - 59 T). This program is aimed particularly at the inspection of very thin and very thick sections.

---

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI on Ultrasonic Testing Procedure (J. C. Smack, chairman) has developed the proposed Method for Ultrasonic Contact Inspection of Weldments appended to this report. The proposed glossary of terms and the procedure for ultrasonic immersion testing are developing quite satisfactorily and should be ready for publication by the Society next year. Other projects include an inspection method using angle projection of the ultrasonic beam by surface contact, reference blocks for ultrasonic inspection of steel, and standardization of ultrasonic instruments and transducers.

The subcommittee recommends the continuation as tentative of Recommended Practices E 113, E 114, and E 127.

Subcommittee VII on Electromagnetic Methods (J. W. Allen, chairman) has appointed four task groups covering respectively nonmagnetic tubing, a glossary of terms, conductivity measurements, and measurement of thickness.

The Task Group on Nonmagnetic Tubing (H. J. Bowman, chairman) is conducting a round-robin series of tests evaluating artificial defects produced in tubing of various analyses and sizes.

The Task Group on Conductivity Measurements (H. Migel, chairman) is reviewing the Method of Test for Electrical Conductivity by Use of Eddy Currents (B 342 - 59 T) developed by Committee B-1 on Wires for Electrical Conductors.

The Task Group on Measurement of Thickness (R. C. Holt, chairman) intends to develop a recommended practice for measuring the thickness of single coating on a single metal by electromagnetic means. Liaison is being established with other ASTM technical committees currently engaged in the measurement of specific types of coating.

This report has been submitted to letter ballot of the committee, which consists of 147 members; 84 members returned their ballots, of whom 82 have voted affirmatively and 0 negatively

Respectfully submitted on behalf of the committee,

J. H. Bly,
Chairman.

A. Gobus,
Secretary.

REPORT OF COMMITTEE E-9

ON

FATIGUE

Committee E-9 held two meetings during the period from May, 1959, to May, 1960: on June 23, 1959, at Atlantic City, N. J., and on Feb. 1, 1960, at Chicago, Ill.

It has become customary to devote the latter part of the E-9 main committee meetings to an informal presentation of a timely fatigue topic. Since a manuscript is not prepared, the topics and speakers are recorded in this report: At the Atlantic City meeting, "Slip Band Cracking in Fatigue of Single Crystals," was presented by W. A. Backofen of Massachusetts Institute of Technology; at the Chicago meeting, "Metallurgical Development of High-Strength Steels and a Discussion of Their Fatigue Properties," by J. P. Sheehan, Armour Research Foundation.

During the past year, three members were elected to the main E-9 Committee. In addition, Minoru Kawamoto, Kyoto University, Kyoto, Japan, was elected to the group of corresponding members. Mr. Kawamoto is chairman of the Committee on Fatigue of JSTM (Japanese Society for Testing Materials) and has published over a hundred papers. The main activity of the corresponding members is exchange of minutes and summaries of programs.

On April 16, 1960, H. F. Moore died at the age of 85. He was a pioneer in the fatigue testing field and made major contributions over a long span of years. The Research Committee on Fatigue, which developed into Committee E-9, was under his leadership for many years. He was a Past-President and Honorary Member of ASTM and an Honorary Member of Committee E-9. On his 75th birthday, the committee held a dinner in his honor. He was truly a great man to whom we shall always be indebted.

In the past year, the committee lost another member in the death of Mr. F. P. Zimmerli. He was currently serving as a consulting member, but in former years was quite active and served the committee very capably.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (T. J. Dolan, chairman).—A summary on "Basic Research in Fatigue of Metals" was prepared following discussion by members of the subcommittee and was published in the Sept. 1959 issue of the ASTM BULLETIN. At the Chicago meeting the discussion topic was applied research as contrasted to earlier discussions on basic research.

Subcommittee II on Papers (D. G. Richards, chairman).—Papers have been reviewed for two sessions on fatigue at the 1960 Annual Meeting. In addition to this, a symposium on Acoustical Fatigue[1] has been developed by B. J. Lazan.

Subcommittee III on Survey (W. S. Hyler, chairman).—During the year, ASTM STP No. 9-J, References on

---

[1] Published as separate publication *ASTM STP No. 284.*

Fatigue 1958, was issued. Work continues on the 10-year index.

Subcommittee IV on Large Machines and Test Correlation (J. F. Millan, chairman).—Recent work was reviewed at the Chicago meeting. Eight topics were covered.

Subcommittee V on Aircraft Structural Fatigue Problems (H. F. Hardrath, chairman).—A symposium on Fatigue of Aircraft Structures[2] was developed for the Third Pacific Area National Meeting at San Francisco held in October, 1959. The symposium covered three sessions and resulted in significant contribution in this important field. Due to the current high level of interest stemming from certain service failures, the subcommittee has been reorganized and expanded considerably.

This report has been submitted to letter ballot of the committee, which consists of 52 members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

---

[2] Published as separate publication *ASTM STP No. 274.*

REPORT OF COMMITTEE E-10
ON
RADIOISOTOPES AND RADIATION EFFECTS*

Committee E-10 on Radioisotopes and Radiation Effects met at San Francisco on Oct. 15, 1959, during the Third Pacific Area National Meeting. Several task forces and subcommittees also met at this time. Another series of meetings was held at Chicago, Ill., in February, 1960 during ASTM Committee Week.

Two symposia were sponsored by Committee E-10 at the Third Pacific Area National Meeting: one on Applied Radiation Radioisotope Test Methods[1] and one on Radiation Effects and Dosimetry.[2] The fifth symposium on Radiation Effects and Dosimetry is to be sponsored by Committee E-10 at the 1960 Annual Meeting.[3]

NEW TENTATIVE

The committee recommends for publication as tentative the Tentative Definitions of Terms Relating to Dosimetry as appended hereto.[4]

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Radiation Induced Changes In Materials (V. P. Calkins, chairman) has organized a number of task groups concerned with metallic materials, ceramic materials, inorganic materials, and organic materials. A dosimetry task group is working on nomenclature, neutron dosimetry in metals, and burnup in fissile materials. Definitions relating to dosimetry are being recommended as tentative and five additional tentatives are being prepared.

Subcommittee III on Tracer Applications (J. L. Kuranz, chairman) has concentrated on developing three of the new tentatives as examples of the versatility and applicability of radioisotopes in ASTM test methods and procedures.

This report has been submitted to letter ballot of the committee, which consists of 195 members; 104 members have returned their ballots, of whom 101 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. E. WEBER,
Chairman.

J. R. BRADFORD,
Secretary.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] To be published as separate publication *ASTM STP No. 268.*

[2] To be included in *ASTM STP No. 276.*

[3] To be published as separate publication *ASTM STP No. 286.*

[4] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 3.

[5] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE E-11

ON

QUALITY CONTROL OF MATERIALS*

Committee E-11 on Quality Control of Materials held four meetings since the last report of the committee: on Nov. 18, 1958; on Mar. 24 and Nov. 18, 1959 and on May 5, 1960, all at ASTM Headquarters in Philadelphia, Pa. The committee's program covers (1) advice to ASTM technical committees on the statistical aspects of standardization problems, and (2) development of recommended practices involving the use of statistical techniques in specific areas of standardization work.

The committee has organized a new Subcommittee II on Bulk Sampling with Louis Tanner as chairman. Scope of the subcommittee which held its organization meeting on May 4, 1960 is:

1. To promote knowledge of statistical theory and methods applicable to the sampling of bulk materials.
2. To advise and assist, when requested, other ASTM committees in planning investigations of the statistical aspects of sampling specific bulk materials, in interpreting the results of such investigations, and in the development of sampling plans for the materials.

A new Task Group on Machine Analysis of Data has been organized with P. S. Olmstead as chairman. The purpose of the project is: (1) to identify the steps in machine analysis of data where recommended practices are needed, and (2) to prepare a recommended practice for the simplest of these steps.

The committee is sponsoring jointly with the Administrative Committee on Research a Symposium on Quality of Observations, to be held at the Sixty-Third Annual Meeting of the Society.

The committee consists of 23 members. The maximum membership authorized by the committee's by-laws is 30.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. Collier.
Vice-Chairman, C. A. Bicking.
Secretary, D. H. W. Allan.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (**E 29 – 58 T**)[1] and continuation of the recommended practices as tentative:

Section 3.—Change Paragraph (*d*) and the referenced Footnote 3 to read as follows:

> (*d*) *Rounding-off Procedure.*—The actual rounding-off procedure[3] shall be as follows:
>
> (*1*) When the figure next beyond the last place to be retained is less than 5, retain unchanged the figure in the last place retained.
>
> (*2*) When the figure next beyond the last place to be retained is greater than 5, increase by 1 the figure in the last place retained.
>
> (*3*) When the figure next beyond the last place to be retained is 5, and

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] 1958 Book of ASTM Standards, Parts 1 to 10.

(*a*) There are no figures, beyond this 5, or only zeros, (i) increase by 1 the figure in the last place retained if it is odd, (ii) leave the figure unchanged if it is even.

(*b*) There are figures beyond this 5, increase by 1 the figure in the last place retained.

This rounding-off procedure may be restated simply as follows: When rounding off a number to one having a specified number of significant places, choose that which is nearest. If two choices are possible, as when the digits dropped are exactly a 5 or a 5 followed only by zeros, choose the one ending in an even digit.

Table I gives examples of applying this round-off procedure.

In Paragraph (*e*), in lines 2 and 3, change ". . . direct rounding off the most . . ." to read ". . . direct rounding-off of the most . . . ."

Change Paragraph (*f*) to read as follows:

(*f*) *Special Case, Rounding-off to the Nearest 50, 5, 0.5, 0.05, etc.*—If in special cases it is desired to specify rounding off to the nearest 50, 5, 0.5, 0.05, etc., this may be done by so indicating in the standard. In order to round off to the nearest 50, 5, 0.5, 0.05, etc., double the observed or calculated value, round off to the nearest 100, 10, 1.0, 0.10, etc., in accordance with the procedure in Paragraph (*d*), and divide by 2. For example, in rounding off 6025 to the nearest 50, 6025 is doubled giving 12,050 which becomes 12,000 when rounded off to the nearest 100 (Paragraph 3(*d*), Item (*3*)(*a*)). When 12,000 is divided by 2, the resulting number, 6000, is the rounded-off value of 6025. In rounding off 6075 to the nearest 50, 6075 is doubled giving 12,150 which becomes 12,200 when rounded off to the nearest 100 (Paragraph 3(*d*), Item (*3*)(*a*)). When 12,200 is divided by 2, the resulting number, 6100, is the rounded off value of 6075.

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—EXAMPLES OF ROUNDING OFF.
(Revision of Table I, Recommended Practice E 29)

Specified Limit	Observed Value or Calculated Value	To be Rounded Off to the Nearest	Rounded-off Value to be Used for Purposes of Determining Conformance	Conforms with the Specified Limit
Tensile Strength, 60 000 psi, min	59 940	100 psi	59 900	No
	59 950	100 psi	60 000	Yes
	59 960	100 psi	60 000	Yes
Nickel, 57 per cent, min	56.4	1 per cent	56	No
	56.5	1 per cent	56	No
	56.6	1 per cent	57	Yes
Water Extract Conductivity, 40 micromhos per cm, max	40.4	1 micromho per cm	40	Yes
	40.5	1 micromho per cm	40	Yes
	40.6	1 micromho per cm	41	No
Sodium Bicarbonate, 0.5 per cent, max	0.54	0.1 per cent	0.5	Yes
	0.55	0.1 per cent	0.6	No
	0.56	0.1 per cent	0.6	No

The recommendation in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF THE COMMITTEE

The committee has in draft form several recommended practices that

---

[3] The rounding-off procedure given in this Recommended Practice is the same as the one given in the American Standard Rules for Rounding Off Numerical Values (ASA No. Z25.1—1940) and in the ASTM Manual on Quality Control of Materials, Part 2, Section 7 (1951).

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

cover specific areas of standardization of importance to the technical committees. For the most part, these drafts are the result of many revisions and rewritings. The committee is working on applications of statistical methods in standardization where these methods have never before been used. In doing so, there is often strong conflict among those on the committee who are expert in the particular field. The resolution of these conflicts is not achieved by cooperative laboratory studies as in the development of standards and specifications for material. Instead, agreement must be reached by study and discussion. The committee is aware of the desire for speed in the development of the practices on which it is working. At the same time, it feels that it is the best policy to recommend action on proposed practices only when there is essentially complete agreement within the committee as to the soundness of the proposals and their theoretical bases.

The following specific projects are active in task groups, with drafts under discussion or being balloted upon:

1. Treatment of Outlying Observations.—The purpose of the project is to prepare a recommended practice on the treatment of outlying observations in numerical data, in a form suitable for use by ASTM members and committees.

2. Smoothing Empirical Data.—The purpose of the project is to prepare a guide for curve fitting in the case of a linear relationship, including a discussion of methods of determining and expressing the limits of uncertainty of the calculated relationship.

3. Precision and Accuracy.—The purpose of the project is to study problems connected with the use of such terms as "Precision," "Accuracy," "Reproducibility," "Repeatability," "Consistency," "Reliability," "Calibration," and other allied terms connected with measurement of error, and as a result make a report with recommendations as to those terms which appear desirable and necessary for standardization. Each term selected for standardization is to be defined precisely and if feasible, a simple algebraic or numerical measure of each term is to be given.

4. Planning Interlaboratory Test Programs.—The purpose of the project is the preparation of recommendations on planning for the collection of multisource data—specifically for aiding committees of the Society.

This report has been submitted to letter ballot of the committee, which consists of 23 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

SIMON COLLIER,
Chairman.

O. P. BECKWITH,
Acting Secretary.

REPORT OF COMMITTEE E-12
ON
APPEARANCE*

Committee E-12 on Appearance met twice in the past twelve months: at Atlantic, City, N. J., on June 24 during the Annual Meeting of the Society, and again at Chicago, Ill. on Feb. 3 and 4, during ASTM Committee Week.

A major contribution was the presentation during the 1959 Annual Meeting of a Symposium on New Visual Aids for Standardizing and Communicating Product Appearance.[1] The symposium contained several excellent papers highlighting selected commercial and industrial areas where measurement and presentation of one or more aspects of appearance can be an important and too frequently an unexploited tool for controlling product quality. Closed-circuit television was revealed as a new technique with undefined potential. Publication of this symposium should expand the range of this stimulus to thought on new uses of factors of appearance.

A panel discussion on "Measurement of Color and Gloss of Anodized Aluminum Alloys" was held at the Chicago meeting. The large attendance reflected the growing awareness of the importance of this problem, the excellence of the programmed phases of the discussion and the aggressive handling of the several facets of this problem by Task Group 4 on Standards for Aluminum Finishes (R. V. Paulson, chairman).

Two new task groups authorized at the Chicago meeting reflect the continuing growth of Committee E-12: Task Group 5 on Standards for Carbon Paper and Typewriter Ribbons under the chairmanship of Mr. W. E. Grady, Carter's Ink Co., and Task Group 6 on Geometric Identification of Reflectance and Transmittance Measurements (R. S. Hunter, chairman). The organization meeting of Task Group 5 in New York, N. Y., on April 27, 1960 was unusually well attended.

A problem challenging Committee E-12 is the continued recruitment of individuals with sufficient initiative and competence to identify, and manage the solution of, important problems related to the appearance of engineering materials.

The officers elected for the ensuing term of two years are as follows:

Chairman, G. W. Ingle.
Vice-Chairman, W. J. Kiernan.
Secretary, S. H. Petry.
Assistant Secretary, Paul Giesecke.

NEW TENTATIVES

The committee recommends for publication as tentative the following recommended practices as appended hereto. These proposals embody comprehensive and authoritative thinking in a relatively unexploited field of measurement.

Tentative Recommended Practice for:

Goniophotometry of Transmitting Objects and Materials,[2] and

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The papers presented at this symposium have been published as *ASTM STP No. 258.*

[2] This new tentative appears in the 1960 Supplement to Book of ASTM Standards, Parts 6 and 8.

Goniophotometry of Reflecting Objects and Materials.[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 78 members; 40 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. W. INGLE,
Chairman.

RICHARD S. HUNTER,
Secretary.

---

[3] This new tentative appears in the 1960 Supplement to Book of ASTM Standards, Parts 6, 8, and 9.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE E-13
ON
ABSORPTION SPECTROSCOPY*

Committee E-13 on Absorption Spectroscopy and most of its subcommittees met twice during the year: in New York City on Nov. 5 and 6, 1959, in conjunction with the Eastern Analytical Symposium, and on Feb. 29 and March 1, 1960, with the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The Pittsburgh Conference included three symposia,[1] one on nuclear magnetic resonance and two on separate phases of molecular fluorescence spectroscopy, organized by members of E-13 subcommittees in these fields.

The officers elected for the ensuing term of two years are as follows:

Chairman, E. J. Rosenbaum.

Vice-Chairman, L. E. Kuentzel.

Secretary, R. F. Robey.

Advisory Committee Members-at-Large: Harlan Foster and R. C. Hirt.

Members of Committee E-13 in western U. S. cooperated with members of Committee E-2 on Emission Spectroscopy in arranging the program for the Symposium on Spectroscopy[2] at the Third Pacific Area National Meeting of the Society, San Francisco, Calif., Oct. 11–16, 1959. This symposium included a paper by R. F. Robey, entitled "Ten Years of Aid to Applied Spectroscopy by Committee E-13," which marks the decennial anniversary of the founding of Committee E-13 and also summarizes and publicizes its activities and accomplishments.[3]

Committee E-13 plans to meet again with the Eastern Analytical Symposium in New York in November, 1960, and with the Pittsburgh Conference in March, 1961.

NEW TENTATIVES

The committee recommends for publication as tentative the following recommended practices:[4]

Tentative Recommended Practice for:

General Techniques of Infrared Quantitative Analysis: As published as information only in *Proceedings*, Am. Soc. Testing Mats., Vol. 59 p. 610 (1959).

General Techniques of Ultraviolet Quantitative Analysis: As published as information only in *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 622 (1959), with the following revisions:

Section 4.—At the end of this section add the following sentences: "The difference between the absorbance of the sample cell and the absorbance of the comparison or blank cell should be determined at each analytical wavelength with both cells filled with solvent. These differences, or cell corrections, should be added algebraically to the absorbance of the sample solution at the proper wavelength."

Section 8.—Paragraph (*a*), at the end of the second subparagraph, in the next to last sentence, reading ". . . . designated as C," add the words: "and is in 'weight per cent' (grams per 100 g)." Change the last sentence to read: "The solution to be examined has a concentration of *sample* in solution, here designated as C_s, which is in units of 'grams per liter.'"

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Copies of the papers are not available except possibly by correspondence with individual authors. Abstracts appear in the official program booklet of the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Feb. 29–March 4, 1960.

[2] To be published as *ASTM STP No. 269.*

[3] Copies of this paper are available at ASTM Headquarters. This paper will not be included in *ASTM STP No. 269*, but will appear in *Applied Spectroscopy.*

[4] The new tentatives appear in the 1960 Supplement to Book of ASTM Standards, Part 7.

In Paragraph (*d*) Item (*3*), to the definition of S add: ", or the ratio of the background absorbance values at these wavelengths." At the end of Item (*3*) add: "The above expression holds only in the special case where the wavelength for A_1 is exactly midway between the wavelengths for A_2 and A_3. The equation for the general case is:

$$C\text{, per cent} = \frac{f\left[A_1 - \left(A_3 + (A_2 - A_3) \times \frac{\lambda_3 - \lambda_1}{\lambda_3 - \lambda_2}\right)\right]}{abC_s} \times 100$$

Add a new Item (*9*) to read as follows:

(*9*) ***Inverted Matrix Method, for Two or More Components with Mutually Overlapping Absorption.***—For information on the inverted matrix method, see Section 7 (*a*) of the Recommended Practices for General Techniques of Infrared Quantitative Analysis (ASTM Designation: E 000) and the literature (**13**).

References.—Add a new reference, as follows: "(**13**) P. D. Crout, "A Short Method for Evaluating Determinants and Solvent Systems of Linear Equations with Real or Complex Coefficients," *Transactions*, Am. Inst. Electrical Engrs., Vol. 60, p. 1235 (1941)."

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Methods (R. T. O'Connor, chairman) prepared the revisions of the Proposed Tentative Recommended Practice for General Techniques of Ultraviolet Quantitative Analysis referred to earlier in this report. Also, this past year the subcommittee reviewed several manuscripts on methods involving absorption spectroscopy prepared by Committees D-1 on Paint, Varnish, Lacquer, and Related Products; D-2 on Petroleum Products and Lubricants; D-12 on Soaps and Other Detergents; and D-16 on Industrial Aromatic Hydrocarbons and Related Materials

Subcommittee III on Standard Data (W. C. Kenyon, chairman) coded hundreds of new spectra during the year for the Wyandotte-ASTM Punched-Card Index to Absorption Spectral Data.[6] The punching operation, transferred to Wyandotte Chemicals Corp. in the middle of the year, produced three supplements to the Card Index from spectra coded by volunteer workers. The Card Index as of Feb. 1, 1960 comprises the following decks:

Spectral Index Deck	Approximate Number of Cards
Infrared	27 000
Ultraviolet	11 000
Infrared Empirical Formula-Name	36 000
Ultraviolet Empirical Formula-Name	16 000
Total	90 000

Decks for visible and near-infrared spectra are in preparation.

Other subcommittees continue to attack the problems in their fields as outlined in the 1959 report of Committee E-13. Many of the problems, such as evaluation of apparatus, nomenclature, and referencing and presentation of spectral data, are at the frontiers of science. Progress of the subcommittees in solving these problems is being followed closely by scientific bodies throughout the world.

This report has been submitted to letter ballot of the committee, which consists of 89 members; 67 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. ROSENBAUM,
Chairman.

R. F. ROBEY,
Secretary.

---

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[6] The Card Index may be purchased from ASTM Headquarters.

REPORT OF COMMITTEE E-14
ON
MASS SPECTROMETRY*

Committee E-14 on Mass Spectrometry held its Eighth Annual Meeting in conjunction with the ASTM Annual Meeting at Atlantic City, N. J., June 26 to July 1, 1960. A registration of 355 made this the second largest meeting held by Committee E-14. A total of 85 papers was presented during the five days; eight countries were represented and four of the invited speakers came from overseas for the meeting. Highlights of the meeting were four symposia: Photoionization, Precise Measurement of Uranium Isotopes, Solids Analysis, and Ion Rearrangement Processes. Contributing to these four symposia were 26 invited speakers. Industrial concerns took part in a mass spectrometry instrument clinic.

Other highlights of the meeting were new instrumental developments and new applications. Among the items of chief interest were four papers and a discussion of five panel members on the mass spectrometry of space.

Three decisions of international importance in mass spectrometry have been made during the past year by the Executive Committee: (1) support was given to A. O. C. Nier's proposal that carbon-12 be adopted as an international atomic weight standard; (2) a second joint conference with the British Institute of Petroleum Mass Spectrometry Panel has been agreed upon and will be held at Oxford University in September 1961. The joint conference in 1958 was a great international success and resulted in a 704-page book, "Advances in Mass Spectrometry," edited by J. D. Waldron and published by Pergamon Press in 1959. At the 1958 meeting Committee E-14 was represented by authors of 15 of the 40 papers. (3) A journal for papers on mass spectrometry has been proposed by a publisher and has been considered by both Committee E-14 and the British Mass Spectrometry Panel. At the Atlantic City meeting, the Executive Committee voted unanimously to oppose the origin of such a journal since too many journals are presently in existence and there is not sufficient difficulty in publishing mass spectrometry papers to justify a new journal. However, it was suggested that journals devoted to molecular spectroscopy only might be encouraged to include papers on mass spectrometry.

The ninth annual meeting on mass spectrometry will be held in Chicago at the Hotel Sherman in June 1961.

The officers and members-at-large elected for the ensuing term of two years are as follows:

Chairman, Vernon H. Dibeler.
Vice-Chairman, Russell E. Fox.
Vice-Chairman, R. A. Brown.
Secretary, George F. Crable.
Members-at-Large, J. H. Beynon and Charles Robinson.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 2 on Theoretical and Fundamental Aspects (J. L. Franklin, chairman) is continuing consideration of the various aspects of theoretical mass spectrometry, making and continuing contacts with overseas workers, preparing and distributing literature surveys on theoretical aspects, and considering symposia subjects to be recommended for Committee E-14 annual meetings.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

Subcommittee 3 on High Molecular Weight Techniques (H. E. Lumpkin, chairman).—A questionnaire was circulated regarding the needs for heavy hydrocarbons and lists of compounds available from the American Petroleum Inst. The form for reporting mass spectra in the American Petroleum Inst. collection was discussed. The new form is based on total ionization. The literature survey was brought up to 1959 and now covers the material from 1950 through 1959. Cooperation has been established with the British group carrying on similar work, and their activities are being coordinated with those of the subcommittee.

Subcommittee 4 on Methods, Data, and Nomenclature (Fred W. McLafferty, chairman) will recommend to the main committee adoption as standard in 1961 of E 137 – 58 T, "Evaluation of Suitability of Mass Spectrometers for Use in Methods of Chemical Analysis." Four other methods originating in Committees D-2 on Petroleum Products and Lubricants and D-3 on Gaseous Fuels were reviewed by the subcommittee. The highly successful cooperative project involving distribution of uncertified mass spectra among cooperating laboratories has resulted in acceptance of the 1030 spectra from contributors. Thus far 666 of these have actually been distributed. The task group handling the data has settled on a coding system for mass spectra and has successfully coded all API mass spectra in cooperation with Wyandotte Chemical Co. The uncertified spectra will next be coded and card files and tabulations will be ready for distribution. A new task group has been formed to consider distribution of a table of exact masses offered by Consolidated Electrodynamics Corp. from measurements on their Mattauch instrument.

Subcommittee 5 on New Instruments and Techniques (Frank W. Karasek, chairman).—The extensive literature survey of the subcommittee has been extended and brought up to date. The meeting of this subcommittee was devoted to an informal discussion on mass spectrometry in space research. The discussion leader and five experts in the field discussed the topics of a mass spectrometer at high altitudes, a magnetic mass spectrometer, studies of micrometeorites, and a new trace-detector for helium.

Subcommittee 7 on Solids Techniques (L. F. Herzog, chairman).—Literature survey work of this subcommittee has been brought up to date. Standardization of solids analyses and data on solids were discussed and will continue to be studied. A talk was given at the meeting on data obtained from spark source instruments.

Respectfully submitted on behalf of the committee,

R. A. Friedel,
Chairman.

Ralph A. Brown,
Secretary.

REPORT OF COMMITTEE E-15
ON
ANALYSIS AND TESTING OF INDUSTRIAL CHEMICALS*

Committee E-15 on Analysis and Testing of Industrial Chemicals was organized Feb. 19, 1959, and has held two meetings: June 26, 1959, in Atlantic City, N. J., and Feb. 4 and 5, 1960, in Chicago, Ill.

Following the organization meeting, a questionnaire was sent to all members on their work interests within the approved scope of the committee. Consideration of the replies by the Executive Subcommittee led to establishment of the following three groups of subcommittees:

General:

- Subcommittee 1, Editorial and Nomenclature (not yet activated)
- Subcommittee 2 on Standards and Reagents
- Subcommittee 3 on Precision and Accuracy
- Subcommittee 4 on Sampling

General Methods:

- Subcommittee A-1 on Chemical Elements
- Subcommittee A-2 on Functional Groups
- Subcommittee A-3 on Physical Properties
- Subcommittee A-4 on Water Determination

Product Methods:

- Subcommittee B-1 on Mineral Acids
- Subcommittee B-2 on Alkalies
- Subcommittee B-3 on Alcohols and Polyalcohols

Chairmen have been appointed for all of the above subcommittees except that for Editorial and Nomenclature, and all are actively working. Responsibilities for the General Methods subcommittees have been assigned to Vice-Chairman E. G. Wiest, and for the Product Methods subcommittees to Vice-Chairman J. T. Woods.

The committee has been assigned the responsibility of Subcommittee 13 on Determination of Water and Subcommittee 15 on Correlation of Chemical Analysis of Committee E-1 on Methods of Testing. It was requested also to consider Committee E-1's Subcommittee 12 on Density, Subcommittee 22 on Hydrogen Ion, and Subcommittee 23 on Volatility, but action has been deferred pending more complete organization of the committee.

Consideration is being given to establishment of working groups on (1) Analysis of Radioactive Chemicals and (2) Gas Chromatography, and to (3) active participation in ISO/TC 47 on Chemicals, particularly on methods of chemical analysis. Product specifications are specifically excluded from the scope of Committee E-15.

Extension of Committee E-15's work will be governed by the interest of its members.

The committee wishes to cooperate with the other committees of the Society and with other societies in developing, coordinating, and reconciling chemical analytical methods wherever it is advantageous to the chemical industry, and does not duplicate existing work.

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

C. M. Gambrill has been appointed a liaison representative for Committee D-2 on Petroleum Products and Lubricants.

C. O. Willits has been appointed a liaison representative for Assn. of Official Agricultural Chemists (AOAC).

R. J. Sobatzki, chairman of Committee E-15's Subcommittee 3 on Precision and Accuracy, has been appointed a liaison representative on Subcommittee I on ASTM Problems of Committee E-11 on Quality Control of Materials.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. A. Kirklin.

Vice-Chairman (General Methods), E. G. Wiest.

Vice-Chairman (Product Methods), J. T. Woods.

Secretary, R. C. Johnson.

Members-at-Large of the Executive Subcommittee: A. Q. Butler and B. H. Cummings.

There are at present 127 members serving on Committee E-15 representing 70 different companies or organizations.

For Publication as Information

The committee recommends for publication as information only the Glossary of Statistical Terms as appended hereto.[1] This glossary is intended as Part A of a Proposed Recommended Practice for Developing Precision Data on ASTM Methods for the Analysis and Testing of Industrial Chemicals, in preparation.

Subcommittee Activities

Subcommittee 2 on Standards and Reagents (C. A. Flanders, chairman) is considering nomenclature and format for instruction on preparation of standard solutions. Two task groups are being set up on (1) standardization of alkali solutions and indicators to be recommended, and (2) standardization of acid solutions and recommended indicators.

Subcommittee 3 on Precision and Accuracy (R. J. Sobatzki, chairman) is preparing the recommended practice for developing precision data mentioned above, the first part of which is the appended glossary.[1]

Subcommittee 4 on Sampling (H. O. Hehner, chairman) has tentatively agreed upon a division of materials into which industrial chemicals may be grouped for sampling purposes and is reviewing existing ASTM and other sampling procedures which may be applicable. Because of the magnitude of the undertaking only the more common sampling problems will be attacked at first. Two task groups have been set upon the bulk sampling of: (1) simple single-phase liquids (R. L. Swallow, leader), and (2) free-flowing finely divided solids (H. O. Hehner, leader).

Subcommittee A-1 on Elements (Leavitt Gard, chairman) has activated a Task Group on Methods for Chlorine and Sulfur in Organic Compounds under the leadership of A. P. Scanzillo, which is making a comprehensive survey of the several different methods commonly used for determining these elements. The most appropriate methods will be selected from this survey for round-robin tests and accuracy studies.

Subcommittee A-2 on Functional Groups (J. Mitchell, Jr., chairman) has activated two task groups on (1) determination of hydroxyl groups in aliphatic organic compounds (T. P. Callen, leader) and (2) determination of unsaturation in organic compounds (E. N. Luce, leader).

The hydroxyl task group is studying the several different acetylation procedures for hydroxyl number now in use and is drawing up a general procedure for cooperative evaluation.

The unsaturation task group has selected the pyridine sulfate dibromide method and the coulometric bromination method for preliminary study on standard samples of methyl methacrylate, maleic acid, kerosine, methyl oleate, and butadiene.

---

[1] See p. 489.

Subcommittee A-3 on Physical Properties (B. H. Cummings, chairman) has been considering the need for uniform density-temperature relationships for industrially pure liquid chemicals. It is planned initially to establish conversion factors whereby a measured volume of a given product at a measured temperature might be accurately converted to a corresponding volume at a temperature commonly used in the laboratory measurement of specific gravity. A Task Group on Specific Gravity (B. H. Cummings, leader) has been activated to develop a method having acceptable precision.

A Task Group on Melting and Freezing Point (or Range) (N. W. MacNaughton, leader) has been activated, also a review of color methods used for industrial chemicals is being made by G. Oplinger for consideration.

Subcommittee A-4 on Water Determination (P. L. Sutton, chairman) selected as its first project the review of various descriptions of the Karl Fischer method. A task group (C. A. Flanders, leader) has been set up to determine the classes of industrial compounds for which it is applicable, types of chemicals that interfere, size of sample for varying amounts of water, and so on, with the aim of drawing up a general procedure. The experiences of other ASTM committees using the Karl Fischer method will be sought through liaison representatives.

Subcommittee B-1 on Mineral Acids (W. Clavan, chairman) has set up two task groups on (1) sulfuric acid (N. C. Schultze, leader) and (2) hydrochloric acid and anhydrous hydrogen chloride (R. E. Richard, leader).

The Task Group on Sulfuric Acid is preparing methods for total acidity of 95 per cent and of fuming types for cooperative evaluation. Members of the committee are being polled to determine what impurities methods should be written, for example, for iron, chloride, nitrate, arsenic, heavy metals, reducing substances, and turbidity.

The Task Group on Hydrochloric Acid is surveying the methods of the industry for evaluation.

Subcommittee B-2 on Alkalies (C. V. Francis, chairman) has prepared proposed methods for total alkalinity, chloride, sulfate, and carbonate in sodium hydroxide, based on the procedures submitted by its various members. These methods are being revised in accordance with suggestions received and will be evaluated in a round-robin program.

Methods for sodium carbonate, sodium bicarbonate, potassium hydroxide, potassium carbonate, and lithium are on the agenda for future consideration.

Subcommittee B-3 on Alcohols and, Polyalcohols (W. R. Dietz, chairman) is considering ethylene glycol and methanol. It is recognized that Committee D-1 on Paint, Varnish, Lacquer, and Related Products has issued both specifications and test methods for methanol used as a solvent. There is no intention to negate or change Committee D-1's test methods, but rather to investigate if additional test methods are needed where methanol is used as a chemical.

Two task groups on (1) ethylene glycol (R. L. Anderson, leader), and (2) methanol (J. L. Carlson, leader) are surveying the membership for present methods of analysis to determine where needs for standardization exists.

This report has been submitted to letter ballot of the committee, which consists of 124 voting members; 59 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. KIRKLIN,
Chairman.

R. C. JOHNSON,
Secretary.

APPENDIX

PROPOSED RECOMMENDED PRACTICE FOR DEVELOPING PRECISION DATA ON ASTM METHODS FOR THE ANALYSIS AND TESTING OF INDUSTRIAL CHEMICALS[1, 2]

This is a proposed recommended practice and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This recommended practice establishes uniform standards for expressing the precision and accuracy of analytical or test methods for industrial chemicals. It includes recommended procedures for the development of precision and accuracy information, based on the simplest elements of statistical design and analysis capable of giving the essential information. These are offered for the guidance of task groups having limited statistical experience, but there is no intent to restrict qualified groups in their use of more advanced designs or other recognized techniques.

(*b*) The various parts appear in the following order:[3]

Part A.—Glossary.

Part B.—Planning the Inter-Laboratory Study.

Part C.—Statistical Design and Analysis.

Part D.—Format of Precision Statements.

Part E.—Accuracy.

Part F.—Presentation of Data.

PART A.—GLOSSARY

Scope

A1. The following statistical terms are defined in the sense in which they will be used in presenting precision and accuracy information. These are stated as simply as possible, so that they can be interpreted and applied by non-statisticians.

Definitions

A2. (*a*) *Error.*—In a statistical sense, any deviation of an observed value from the expected or true value. When reported in the same units as those used for the measurement, the error is termed *absolute*. When expressed as a fraction or percentage of the value measured, it is called a *relative* error. All statements of precision and accuracy should indicate clearly whether they are expressed in an absolute or relative sense.

(*b*) *Random Error.*—The chance variation encountered in all experimental work despite the closest possible control of recognized variables. It is characterized by the random occurrence of both positive and negative deviations from the expected or true value, the algebraic sum of which will approach zero in a long series of measurements. Errors of this type are often referred to as *indeterminate errors*.

(*c*) *Bias.*—A constant or systematic error as opposed to a random error.

---

[1] This proposed recommended practice is under the jurisdiction of the ASTM Committee E-15 on Analysis and Testing of Industrial Chemicals.

[2] Published as information, June, 1960.

[3] At the present time only Part A, Glossary, has been completed. Parts B to F are still in preparation and will be published when available.

It manifests itself as a persistent positive or negative deviation from the expected or true value. Errors of this type are often referred to as *determinate* errors.

(*d*) *Precision.*—The degree of agreement of repeated measurements of the same property expressed in terms of dispersion of test results about their average. Precision statements in ASTM methods for industrial chemicals will be based on the standard deviation of a series of measurements and will be stated in terms of the repeatability and reproducibility of the method.

(*e*) *Accuracy.*—Although the term "accuracy" is frequently used in chemical work to express the agreement between an experimentally determined mean value and the true or expected value, in ASTM usage this term assumes a broader meaning combining the concepts of both precision and bias. To avoid confusion, the term "bias" will be used to appraise the systematic error of test methods for industrial chemicals.

(*f*) *Variance.*—A measure of the dispersion of a series of results around their average. It is calculated by summing the squares of the individual deviations from the average of the results and dividing by the number of results minus one.

(*g*) *Standard Deviation.*—A measure of the dispersion of a series of results around their average, expressed as the square root of the quantity obtained by summing the squares of the deviations from the average of the results and dividing by the number of observations minus one. It is also the square root of the variance and may be calculated as follows:

$$s = \sqrt{\frac{\Sigma(X_i - \bar{X})^2}{n - 1}}$$

where:

s = standard deviation of the series of results,
X_i = each individual value,
$\bar{X}$ = average (arithmetic mean) of all values, and
n = number of values.

(*h*) *Coefficient of Variation.*—A measure of relative precision calculated as the standard deviation of a series of values divided by their average. It is usually multiplied by 100 and expressed as a percentage.

(*i*) *95 per cent Confidence Interval or Confidence Limits.*—That interval or range of values which will, in 95 per cent of the cases, include the true or expected value. The true or expected value is defined as one calculated from a long or infinite series of determinations.

(*j*) *95 per cent Confidence Level.*—This term is commonly used in establishing the probability of precision statements and means that there are 19 in 20 chances of being correct, and 1 in 20 chances of being wrong, in predicting that the expected precision (or expected value) will fall within the specified limits or range.

(*k*) *Repeatability.*—An estimate of the precision of a method expressed in terms of the agreement attainable between determinations performed by a single analyst using the same apparatus and techniques.

(*l*) *Reproducibility.*—The precision of a method expressed in terms of the agreement attainable between determinations performed in different laboratories, using similar equipment and techniques.

(*m*) *Duplicates.*—Paired determinations performed by one analyst at essentially the same time.

(*n*) *Replicates.*—Two or more independent determinations performed by different analysts, or by the same analysts at different times.

PARTS B TO F

(Parts B to F are still in preparation and will be published when available.)

REPORT OF COMMITTEE F-1
ON
MATERIALS FOR ELECTRON TUBES AND SEMICONDUCTOR DEVICES*

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices held three meetings during the year: in Skytop, Pa., Nov. 5 and 6, 1959; in Washington, D. C., Feb. 25 and 26, 1960; and in Boston, Mass., June 16 and 17, 1960. The meetings were well attended, averaging 209 members and guests per meeting.

The committee membership totals 84, of whom 40 are classified as producers, 33 as consumers and 11 as general interest members.

Stanton Umbreit resigned as Secretary, due to retirement. The committee is pleased to take cognizance of Mr. Umbreit's long service and valuable contributions to the committee's work.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. A. Standing.
Vice-Chairman, F. J. Biondi.
Secretary, C. L. Guettel.
Assistant Secretary, G. W. Andeweg.

J. C. Hickle was appointed chairman of Subcommittee IX, Materials Analysis, replacing R. S. Kelly.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1959 Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the recommendation for publication as tentative of Specifications for Disk Cathode Assemblies. The Specifications are still before the Standards Committee.

NEW TENTATIVE

The committee recommends for publication as tentative the Recommended Practice for Testing Electron Tube Parts Using a Reference Planar Diode as appended hereto.[1]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Volatile Content of Germanium Oxide (**F 5 - 58 T**),[2]
Bulk Density of Germanium Dioxide (**F 6 - 58 T**),[2]
Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (**F 205 - 58 T**),[3] and
Sag of Tungsten Wire (**F 269 - 58 T**).[3]

Tentative Specification for:

Molybdenum Wire Under 20 Mils in Diameter (**F 289 - 56 T**).[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

---

* Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The new tentative appears in the 1960 Supplement to Book of ASTM Standards, Part 2.

[2] 1958 Book of ASTM Standards, Part 2.

[3] 1959 Supplement to Book of ASTM Standards, Part 2.

Tentative Specification for:

- Clear Nickel-Clad and Nickel-Plated Steel Strip for Electron Tubes (**F 1 - 57 T**),
- Aluminum-Clad Steel Strip for Nickel-Steel-Aluminum Composite Strip for Electron Tubes (**F 2 - 57 T**),
- Clear Nickel Strip for Electron Tubes (**F 3 - 57 T**),
- Carbonized Nickel Strip and Carbonized Nickel-Plated and Nickel-Clad Steel Strip for Electron Tubes (**F 4 - 57 T**),
- Aluminum Oxide Powder (**F 7 - 58 T**),
- Nickel Alloy Cathode Sleeves for Electron Devices (**F 239 - 57 T**),
- Tungsten Wire for Electronic Devices and Lamps (**F 288 - 57 T**), and
- Round Wire for Use as Grid Lateral Winding Wire in Electron Tube Grids (**F 290 - 57 T**).

Tentative Method of Test for:

- Testing Sleeves and Tubing for Radio Tube Cathodes (**F 128 - 56 T**),
- Sublimation Characteristics of Metallic Materials by Electrical Resistance (**F 278 - 55 T**), and
- Interface Impedance Characteristics of Vacuum Tube Cathodes (**F 300 - 57 T**).

Tentative Recommended Practice for:

- Testing Electron Tube Materials Using a Reference Triode (**F 8 - 58 T**), and
- Cathode Melt Prove-in Testing (**F 238 - 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Cathode Materials (A. P. Haase, chairman) worked principally with improved methods for materials evaluation in the form of a planar triode, with plans to incorporate new features into Specification F 278. Prominent in the program were studies on temperature measurements, featuring comparisons of optical, infrared, thermocouple resistance, and retarding field methods. In considering the studies on interface impedance, the preparation of the history of ASTM activities toward solution of this problem was reviewed with an aim toward publication. A paper on pulse triode method for measuring interface resistance was given by Mr. Stacy. Collapse strength of cathode sleeves received attention, and improvements in the present method were indicated using standard chromium-plated loading pins. Improved accuracy of measuring cathode sleeve dimensions likewise has been under consideration, with studies aimed at improved methods of test for adherence with an approach via heat cycling, interface impedance, and scratch test. An improved system for evaluation of cathode melts was put into effect. Melt No. 102 received particular attention in this regard.

The subcommittee is reviewing tentative Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (F 128 - 56 T), Recommended Practice for Cathode Melt Prove-in Testing (F 238 - 57 T), Specification for Nickel Alloy Cathode Sleeves for Electronic Devices (F 239 - 55 T), and Method of Test for Sublimation Characteristics of Metallic Materials by Electrical Resistance (F 278 - 55 T).

Subcommittee II on Insulators (C. M. Harmon, chairman) was mainly concerned during the year with (1) the preparation of a suitable specification for mica bridges used in electron tubes, considering in this effort five thickness ranges. It is expected that this specification will satisfy certain recommendations brought to the subcommittee's attention by the JEDEC committees. (2) A review of muscovite black mica, particularly visual properties for electron tube micas, was made in coordination with Committee D-9 on Electrical Insulating Materials. (3) A study of aluminum oxide powder in the form of a review of Specification F 7 was conducted. Particular attention was paid to "water solubles" in alumina. This review was conducted with special cognizance of the expanding use of ceramic parts in electron tube making.

---

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Strip Materials (P. P. Pritchett, chairman) was involved in (1) studies of gas in metal strip, (2) preparation of a specification for plate material of high thermal conductivity, (3) consideration of aluminum deoxidized steel with reference to present specifications, and (4) studies of measurements of strip material to closer tolerances.

Subcommittee IV on Wire (D. R. Kerstetter, chairman).—Activities for the year highlighted the following: (1) review of specifications and methods of test for fine tungsten wire, pointing up certain aims toward standard wire sizes, methods other than weight for evaluating outside diameter, effects of anvil sizes and pressures, etc.; (2) evaluation of pull testers for lead wires. This effort is a part of the general review of specifications on miniature lead wires. (3) Consideration of adding finer-sized permanickel and tungsten wires to Specification F 290; (4) consideration of testing machine parameters, such as speed of test, etc.; and (5) general review and revision of all specifications under the committee's jurisdiction on wire, including the following: (*a*) initiation of Specification F 113; (*b*) withdrawal of Specification F 157 which was incorporated in Specification F 9; and (*c*) withdrawal of Specification F 268 which was incorporated in Specification F 9.

Subcommittee V on Metallic and Nonmetallic Seals (V. J. DeSantis, chairman).—Considerable emphasis during the year was placed on the following: (1) establishing specifications for nickel-iron-cobalt alloys; (2) consideration of specifications on nickel-iron alloys having compositions of 42, 46, and 52 per cent nickel; (3) evaluation of expansivity at temperatures above 1000 C (this effort is being coordinated with Committees B-4 on Materials for Thermostats and for Electrical Resistance, Heating and Contacts, C-14 on Glass and Glass Products, and E-1 on Methods of Testing); (4) establishing a standard test piece for metal-ceramic seals, together with standard seals in glass and ceramic; and (5) attention to the color of dumet as it pertains to glass sealing.

Subcommittee VI on Semiconductors (H. M. Pollack, chairman) established study groups on subjects which would provide more adequate background by establishing better methods of test and specifications, essentially on the following: (1) growth of silicon crystals, (2) lifetime of minority carriers, (3) furnace construction, (4) oxygen content of silicon, and (5) intermetallic semiconductors.

Appropriate task forces have been set up to deal with the various important aspects. Appropriate time was also spent on the method of measurement of electrical resistivity. This is particularly a "non-contact" test.

Subcommittee VIII, Editorial (E. A. Thurber, chairman) has monitored all specifications originating in the committee over the year, most recently having reviewed and completed the following: (1) Proposed Tentative Methods of Test for Resistivity of Semiconductor Materials, (2) Proposed Tentative Method for Growing Monocrystalline Test Ingots of Silicon, (3) revision of Tentative Recommended Practice for Testing Electron Tube Materials Using a Reference Triode (F 8), and (4) revision of Proposed Revision of Tentative Recommended Practice for Cathode Melt Prove-in Testing (F 238).

In addition, the Editorial Committee is reviewing specifications with an eye toward incorporating both metric and

English systems of measurement where feasible.

Subcommittee IX on Materials Analysis (J. C. Hickle, chairman).—The function of this subcommittee is largely to provide a specialized service group in the field of analysis to the other subcommittees. During the past year they have highlighted the following: (1) methods of test to determine sulfur in nickel (standardizing on National Bureau of Standards sample 169); (2) proposed methods for quantitative analysis of aluminum oxide, which program is now in the field laboratory testing stage; (3) recommending a neocuproine method for copper as a substitute for the hydrobromic acid method; (4) wet chemical analysis of glass-to-metal sealing alloys; and (5) a general strengthening of the liaison activities with Committees E-2 on Emission Spectroscopy and E-3 on Chemical Analysis of Metals.

Subcommittee X on Contaminants (D. E. Koontz, chairman).— The period since the last annual report has largely been concerned with completing the organization of the group. The work includes: (1) development of test methods for processing materials; (2) evaluating contaminants on component surfaces. Work is aimed at methods for identification of clean metal or ceramic surfaces and contaminants arriving via air or liquid. (3) Extensive literature searches have been completed and appropriate task groups set up to deal with the problems; and (4) most recently a section to deal with sorbed gases and inorganic ions has been formed.

This report has been submitted to letter ballot of the committee, which consists of 84 members; 75 members returned their ballots, of whom 64 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
Chairman.

C. L. GUETTEL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the recommendation that Specifications for Mica Bridges for Electron Tubes be published as tentative. This recommendation was accepted by the Standards Committee on September 29, 1960, and the new tentative specifications appear in the 1960 Supplement to Book of ASTM Standards, Part 2, bearing the designation F 12 – 60 T.

REPORT OF COMMITTEE F-2
ON
FLEXIBLE BARRIER MATERIALS

Committee F-2 held four meetings during the past two years: on October 15, 1958, in Chicago, Ill.; on April 15, 1959, in Chicago, Ill.; on November 19, 1959, in New York, N. Y.; and on April 7, 1960, in Atlantic City, N. J. Special meetings of the Advisory Committee were also held on July 16, 1958, in Tarrytown, N. Y.; on March 4, 1959, in Clifton, N. J; and on September 30, 1959, in Pittsburgh, Pa.

The importance of liaison activities between Committee F-2 and other committees in the Society, as well as with outside organizations, resulted in the organization of Subcommittee V on Liaison.

The officers elected for the ensuing term of two years are as follows:

Chairman, C. C. Sutton.
Vice-Chairman, L. F. Swec.
Secretary, R. H. Giljohann.
Membership Secretary, H. E. Lockhart.
Members-at-Large, W. B. Tibbets and M. Wagner.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Executive Subcommittee (C. C. Sutton, chairman) has made contact with the Food and Drug Administration, U.S.D.A., in order to explore the development of standard methods for determining extractibles from flexible barrier materials. A brochure giving the committee structure, subcommittee scopes, by-laws, and membership is in preparation for publicity purposes.

Subcommittee II on Nomenclature (H. H. Haines, chairman) has circulated four definitions and is working on eight additional definitions of terms important to the flexible barrier industry.

Subcommittee III on Test Methods (K. W. Ninnemann, chairman) has formed three sections with work covering various aspects of testing in this field.

The section on transmission properties has collected and is evaluating ten different gas transmission methods as used by 38 organizations. Methods currently used in the industry for water vapor transmission are also being evaluated. Transmission of other liquids and gases are also under consideration.

The section on mechanical or physical properties has prepared a method for the seal strength of flexible barrier materials. Methods for tear, impact, and bursting strength are being developed.

The section on chemical properties is developing a method for determining the moisture content of flexible barriers.

Subcommittee IV on Specifications (Paul Cope, chairman) is determining all of the properties which must be defined in order to specify a flexible barrier. Existing test methods for each of these properties are being collected and evaluated in order to assist in defining the long-range program of Subcommittee III.

Subcommittee V on Liaison (J. W. Goff, chairman) has established liaison with six ASTM technical committees and is actively following the work currently being carried out by other organizations in this field.

Respectfully submitted on behalf of the committee,

C. C. SUTTON,
Chairman.

T. M. HILL,
Secretary.

TECHNICAL PAPERS

With Discussions

SUMMARY OF PROCEEDINGS OF SESSIONS DEALING WITH PROPERTIES OF CRYSTALLINE SOLIDS

Symposium on Recent Progress in Materials Sciences
and
Symposium on Nature and Origin of Strength of Materials

These Symposia represent the first public effort of the Society's Division of Materials Sciences. These two broad-subject symposia were presented at the first and second sessions of the Sixty-third Annual Meeting of the Society held on June 27, 1960. The two symposia entitled "Recent Progress in Materials Sciences" and "Nature and Origin of Strength of Materials" have been combined under the title "Properties of Crystalline Solids," as all the papers deal with this subject.

Mr. I. V. Williams, Bell Telephone Laboratories, acted as chairman of the first session. Vice-President A. A. Bates, Portland Cement Association, and Mr. J. D. Sullivan, Battelle Memorial Institute, acted as co-chairmen of the second session.

In the introductory paper, which was presented as a Luncheon Address between the two sessions, W. O. Baker, vice-president of research, Bell Telephone Laboratories places in perspective the Society's role in materials sciences as a part of its long standing function of promoting materials knowledge. According to Dr. Baker—

> "All during these heroic times of science and technology, the studying, using, inventing, and testing of materials went on, but they were hardly considered to be domains suitable for detached thought and for the application of bold hypotheses and brilliant theory. In recent decades this situation has changed dramatically. The names who helped to change it have been mentioned in the papers of today's symposia. It is an historic event in the evolution of science and technology to hold symposia like these, with speakers of such vision. This, along with the establishment of the Division of Materials Sciences of ASTM, signifies that bulk properties of matter do challenge the deepest understanding as well as the best exploitation. We have been excited to see the nucleation and growth of the new Division, an epitaxial growth on the firm base of Professor Woods and his Steering Committee, and particularly the undislocated, non-vacant lattice points of co-chairmen J. H. Koenig and I. V. Williams, A. A. Bates and J. D. Sullivan."

Continuing, Dr. Baker emphasized the dimensions of the new domain that is being established by the joining of materials technology, in which ASTM has been so vital a force, and the science of solids constituted of the physics of crystals, mechanics, chemistry, metallurgy, and of many diverse resources of mathematics, statistics, electronics and certainly of dialectics. Research and development of materials, he said, offer both the task and the chance for ventures of the mind as rewarding and revealing as those of the great epics of physical science—the wave-particle duality of radiation and electronics, the composition and formation of the nucleus, the spectroscopy of the separate atom and the molecule, the chemistry of combination and structure.

The following papers are included in the publication:

Luncheon Address

The National Role of Materials Research and Development—W. O. Baker

Symposium on Recent Progress in Materials Sciences

Accomplishments and Limitations of Solid State Theory—Harvey Brooks
The Influence of Surfaces on the Properties of Materials—M. J. Sinnott
Mechanical Properties of Semiconductors—John N. Hobstetter
Status of Ductile Ceramic Research—Earl R. Parker

Symposium on Nature and Origin of Strength of Materials

Dislocation Motions and the Yield Strengths of Solids—John J. Gilman
Resistance to Creep Deformation and Fracture in Metals and Alloys—F. Garofalo
Brittle Fracture Strength of Metals—E. T. Wessel
Effects of Size and Shape on Fracture of Solids—G. R. Irwin
Fatigue Strength of Crystalline Solids—G. M. Sinclair and C. E. Feltner

These papers were issued as *ASTM Special Technical Publication No. 283*, entitled "Properties of Crystalline Solids."

FATIGUE PROPERTIES OF SOME HIGH-STRENGTH STEELS*

By H. E. Frankel,[1] J. A. Bennett,[2] and C. M. Carman[3]

Synopsis

The rotating-beam fatigue properties of 18 high-strength low-alloy steels were investigated. Except for the high-silicon steels, the maximum fatigue strength was generally obtained for the lowest tempering temperature. The steels containing 1.3 per cent copper had higher maximum fatigue strengths than any of the other materials. It is suggested that the generally superior fatigue properties of the copper-bearing materials may be due to the precipitation of copper under the influence of the fluctuating stress.

There is widespread interest in steels having useable tensile strengths above 200,000 psi. In connection with a study of the possible use of materials of this type for ordnance applications, the National Bureau of Standards was requested by the Frankford Arsenal to determine the fatigue properties of certain high-strength steels that are in current production as well as a number of experimental compositions.

The primary purposes of this investigation were to obtain more complete fatigue data on the commercial steels than are available in the literature and to evaluate the fatigue strengths of the experimental steels. While the results of this investigation cannot be considered as wholly conclusive, the trends seem worthy of presentation.

Materials and Heat Treatment

The compositions and premachining histories of the steels are given in Table I.

The rough-machined specimens of each steel were heat treated as a group, in a muffle furnace using a cracked ammonia-propane gas atmosphere, at an appropriate austenitizing temperature. After quenching in oil, the specimens were tempered for 4 hr in a circulating air furnace at the temperatures indicated in Table II.

After heat treatment, the fatigue specimens were ground with a 7-in. diameter wheel, the plane of which was parallel to the length of the specimen. This operation was carefully controlled to avoid overheating the surface, the final cuts being very shallow. The preparation of the specimens was completed by polishing in a longitudinal direction with 1 G paper followed by 400 Aloxite paper. These operations were carried out on the automatic polishing machine described several years ago (1). This device operates at low speed with a small constant pressure between the surface and the

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] National Aeronautics and Space Administration, Goddard Space Flight Center, Greenbelt, Md.

[2] Chief, Mechanical Metallurgy Section, National Bureau of Standards, Washington, D.C.

[3] Metallurgist, Thermal Metallurgy Branch, Pitman-Dunn Laboratories, Frankford Arsenal, Philadelphia, Pa.

abrasive. Although this procedure does not produce a highly polished surface, the surface condition is reproducible from specimen to specimen. All specimens were coated with a rust-preventive oil immediately after polishing. The oil coating was renewed immediately before testing.

TABLE I.—CHEMICAL COMPOSITION, PER CENT BY WEIGHT.

Steel	Carbon	Manganese	Silicon	Chromium	Nickel	Molybdenum	Vanadium	Copper
No. 1	0.40	0.71	0.33	0.81	1.67	0.25	0.15	...
No. 2	0.31	0.91	0.31	0.89	1.86	0.41	0.07	...
No. 3	0.33	0.91	1.63	0.81	1.66	0.42	0.07	...
No. 4	0.25	1.38	1.48	0.05	1.80	0.42	...	...
No. 5	0.27	1.41	1.50	0.34	1.78	0.44	...	...
No. 6	0.43	0.82	1.51	0.73	0.84	0.48	0.07	...
No. 7	0.42	0.85	1.49	1.00	2.01	0.50	0.11	...
No. 8	0.45	1.35	0.15	0.77	0.03	0.68	0.21	0.09
No. 9	0.45	1.33	0.38	0.80	0.02	0.69	0.21	0.74
No. 10	0.45	1.40	0.17	0.83	0.06	0.68	0.17	1.28
No. 10A	0.48	1.18	0.19	0.82	0.05	0.66	0.17	1.32
No. 11	0.46	1.42	0.23	0.86	0.03	0.68	0.22	2.13
No. 12	0.45	0.53	0.14	0.78	0.04	0.66	0.21	0.74
No. 13	0.46	0.52	0.20	0.79	0.05	0.66	0.21	1.28
No. 14	0.49	0.52	0.16	0.78	0.05	0.65	0.20	2.06
No. 15	0.31	1.54	0.21	0.83	0.06	0.67	0.19	1.29
No. 16	0.32	1.49	0.20	0.84	0.06	1.04	0.18	1.28
No. 17	0.26	1.22	0.12	0.85	0.06	0.68	0.18	1.28

NOTE.—Prior history of the steels:

No. 1......A vanadium-modified AISI 4340, taken from a gun chamber made by upsetting seamless tubing.

No. 2......A vanadium-modified steel of AISI type 4330, product of a 2000-lb induction heat rolled to 1-in. round.

No. 3......Experimental 500-lb induction heat press forged to 6-in. square from 13-in. ingot. Hammer forged to $\frac{1}{2}$-in. square.

No. 4......Vacuum melted.

No. 5......Received as an 8-in. round and forged to 1-in. square for specimens.

No. 6......Product of a 7-ton electric-furnace heat forged to $1\frac{1}{2}$-in. square for specimens.

No. 7......100-lb induction heats, rolled to 1-in. diameter rounds for specimens.

No. 8–17...Experimental steels, 100-lb induction heats, rolled to 1-in. diameter rounds for specimens.

TESTING PROCEDURE

Tensile Properties

The static mechanical properties were determined at the Frankford Arsenal. The tensile properties of steels having yield strengths less than 200,000 psi were determined by means of standard 0.505-in. diameter specimens and conventional autographic stress-strain recording equipment. For materials having yield strengths greater than 200,000 psi, a 0.357-in. diameter, button-head specimen was used. This type of specimen was tested in a special concentric fixture based on a design of Sachs et al (2). All tension tests were made in duplicate; the average results are presented in Table II. The hardness values reported in Table II are the averages of seven determinations.

Fatigue Tests

Rotating-beam fatigue tests were conducted on R. R. Moore machines, using at least eight specimens of each steel for each tempered condition. To avoid excessive heating at the high stress amplitudes used, the machines were operated at speeds of 1000 to 2000 rpm. The stress

TABLE II.—TENSILE AND FATIGUE PROPERTIES.

Steel	Tempering Temperature, deg Fahr	Rockwell Hardness, C Scale	Tensile Strength, psi	Yield Strength, psi[a]	Fatigue Strength, at 10^5 Cycles, psi
No. 1	400	53	276 000	208 500	<140 000
	600	49	253 600	227 000	<130 000
No. 2	400	49.5	252 000	186 000	143 000
	600	46	231 000	191 000	159 000
No. 3	400	51	257 500	190 500	136 000
	600	48.5	241 000	192 800	134 000
No. 4	400	47.5	232 900	165 200	166 000
	600	46.5	225 100	177 400	164 000
No. 5	400	50	241 800	185 800	140 000
	600	49	235 400	194 000	140 000
No. 6	450	54	303 600	246 100	140 000
	650	51.5	291 400	249 100	150 000
No. 7	400	54.5	301 000	216 600	168 000
	600	54	285 200	226 000	159 000
No. 8	350	53	283 000	196 000	175 000
	450	51	277 000	224 000	170 000
	600	49	252 000	221 000	153 000
No. 9	350	53.5	290 000	191 000	169 000
	450	51	273 000	218 000	157 000
	600	49	225 000	218 000	139 000
No. 10	400	52	295 000	230 000	201 000
No. 10A	350	55	303 250	193 500	182 000
	400	52.5	290 000	210 500	175 000
	450	51.5	282 500	224 000	164 000
	600	49.5	260 500	219 450	152 000
No. 11	350	54	309 000	188 000	177 000
	450	52	291 000	222 000	157 000
	600	50	264 500	221 000	139 000
No. 12	350	53	294 000	206 000	165 000
	450	51.5	265 000	230 000	149 000
	600	50	254 000	228 000	140 000
No. 13	350	53.5	238 000	212 000	189 000
	450	52	264 500	229 000	165 000
	600	49	236 000	222 000	148 000
No. 14	350	54	292 000	194 000	174 000
	450	52	283 000	223 000	164 000
	600	49	261 000	215 000	147 000
No. 15	400	50	251 000	185 000	180 000
No. 16	400	49.5	255 000	196 000	185 000
No. 17	400	47	237 500	183 800	180 000

[a] Based on 0.1 per cent offset.

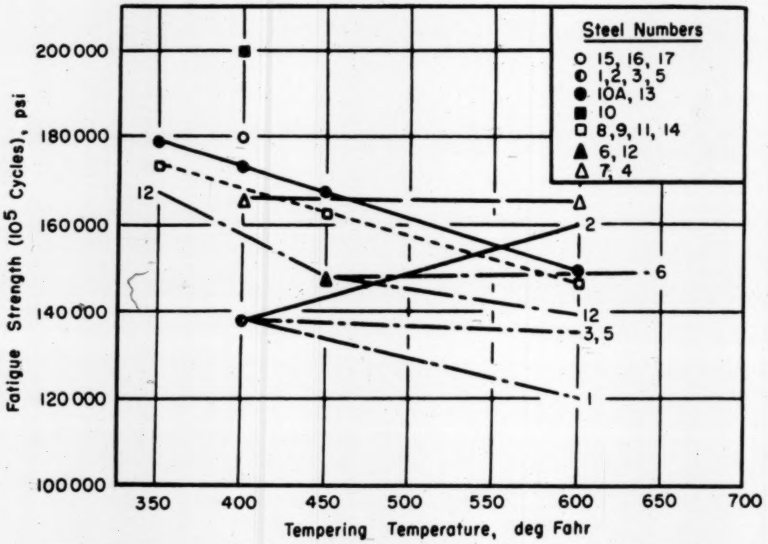


FIG. 1.—Fatigue Strengths at 10^5 Cycles of All Steels Tested, as Influenced by Tempering Temperature.

Steels 10, 10A, 13, 15, 16 and 17 contain 1.3 per cent copper. Steels numbered 3 to 7 have silicon contents of approximately 1.5 per cent. The numbers at the ends of the divergent lines represent plotted points (see Table II).

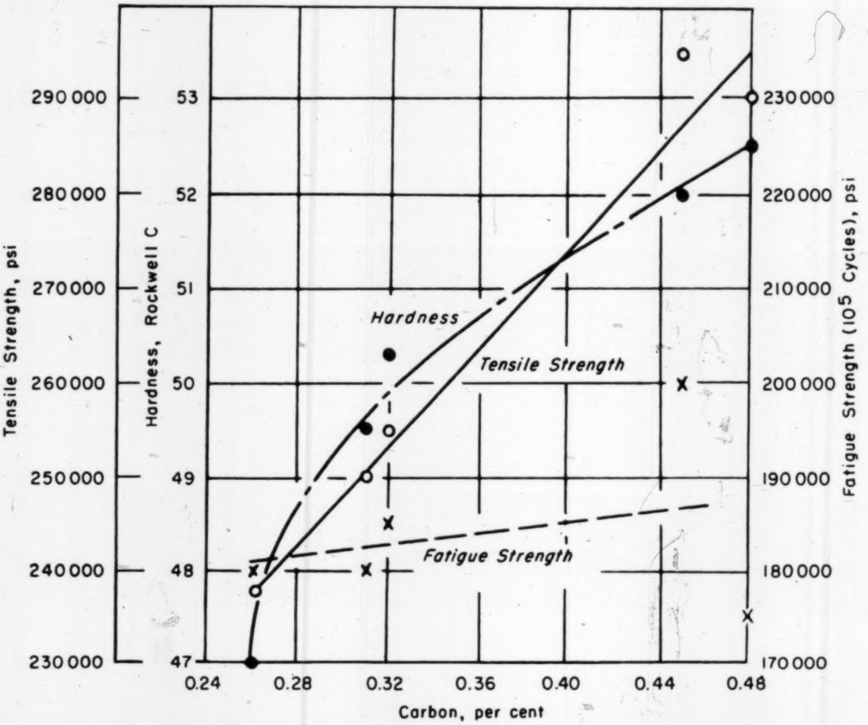


FIG. 2.—Tensile Strength, Hardness and Fatigue Strength at 10^5 Cycles as a Function of Carbon Content.

The values used are those for steels 10, 10A, 15, 16, and 17 tempered at 400 F.

levels were chosen so as to provide information on the fatigue strengths between 10^4 and 10^5 cycles. No attempt was made to determine the fatigue limit.

RESULTS AND DISCUSSION

In order to extract as much information as possible from the results, two approaches were used in analyzing the data. The first was to compare all the data on the basis of some common condition, such as tempering temperature. The second was to compare those steels whose compositions were similar, with perhaps but one element varied.

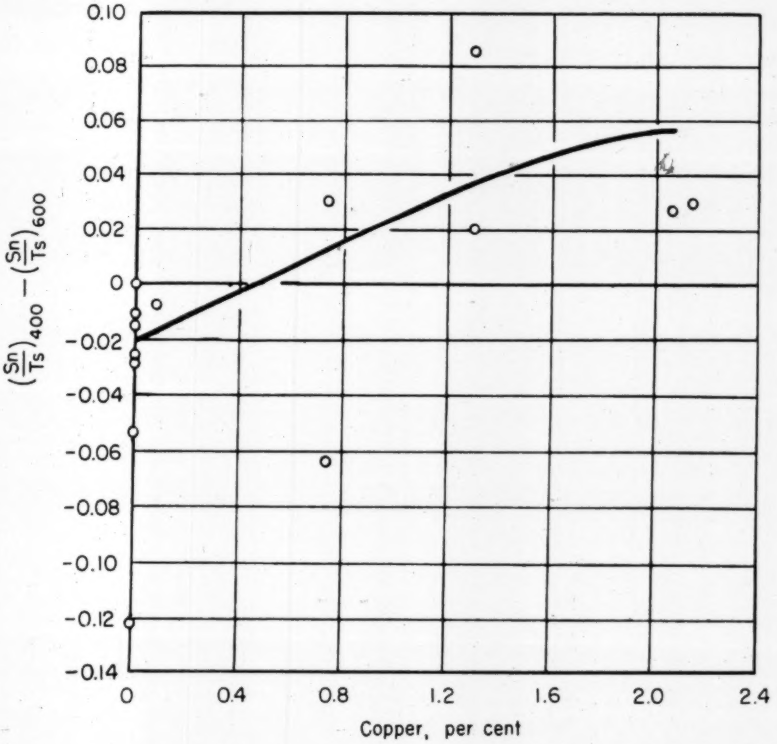


FIG. 3.—The Influence of Tempering Temperature on the Fatigue Strength–Tensile Strength Ratio as a Function of Copper Content.

All of the experimentally obtained values were plotted on conventional S - log N coordinates. Since the fatigue strength for 3×10^4 cycles or less was greater than the yield strength in a number of cases, all comparisons of fatigue strengths are based on the intercepts of the S-N curves at 10^5 cycles. Table II shows the fatigue strength of each steel as a function of tempering temperature. There were usually not more than two or three specimens at any one stress level, so no attempt was made to evaluate the dispersion of the data. When the graphs for the different steels at a particular tempering temperature were compared it became apparent that certain steels could be grouped together, since the differences between them were too small to be significant. The data for all the specimens in a group were therefore plotted together and the best value of the fatigue strength at 10^5 cycles determined. Figure 1 shows this value of fatigue strength for each group as a function of tempering temperature. Data were also obtained with some of the steels tempered at about 1000 F, but because of the relatively low fatigue strengths observed, the results are not presented.

With one exception (No. 2), all of the steels having normal silicon contents, that is, silicon less than about 0.4 per cent, showed an increase of fatigue strength with decreasing tempering temperature. The maximum fatigue strengths reached by these steels was obtained at the lowest tempering temperature used, either 350 or 400 F. The fatigue strengths of those steels containing a greater than normal amount of silicon remained essentially constant with increasing tempering temperature, at least up to about 600 to 650 F. All of the steels containing approximately 1.3 per cent copper, when tempered at 400 F or lower, showed higher maximum fatigue strengths than any others.

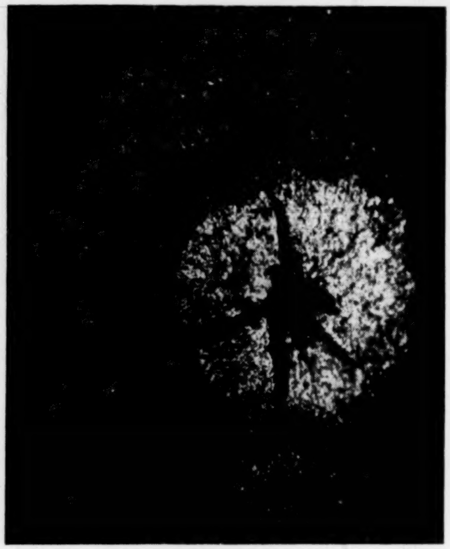


FIG. 4.—Initiation of a Crack Below the Surface. This was typical of steels 10, 15, 16 and 17. (× 200). Reduced to three-quarter size for publication.

Frankel, Bennett, and Pennington (3) have shown that as carbon content is increased a higher hardness is necessary for attaining equivalent fatigue strengths. Although the specimen preparation procedures in the current investigation were not the same as in the earlier work, this general tendency was verified. Figure 2 shows the hardness, tensile strength, and fatigue strength for steels 10, 10A, 15, 16, and 17, which had similar compositions except for the carbon content, and also in the case of 16, molybdenum content. For a constant tempering temperature, the higher-carbon steels had higher tensile strengths and hardnesses, but only slightly higher average fatigue strengths.

There appeared to be a consistent difference between the copper and the noncopper steels. This difference is made more apparent by plotting the behavior of the differences between the fatigue strength - tensile strength ratio when the material had been tempered at 400 F and the ratio when the steel was tempered at 600 F, that is, $\left(\frac{Sn}{Ts}\right)_{400} - \left(\frac{Sn}{Ts}\right)_{600}$, as a function of per cent copper, Fig. 3. This curve includes all the steels that were tempered between 350 and 600 F, except for No. 4 which had a fatigue strength - yield strength ratio greater than 1. The $\left(\frac{Sn}{Ts}\right)_{400}$ ratios for some steels not tempered at 400 F were obtained by interpolation. The ratio differences for all steels containing more than 0.74 per cent copper were positive, while those for the alloys containing zero per cent copper were negative.

Certain of the steels showed a marked tendency for subsurface initiation of the fatigue crack. Of the 32 specimens of steels 10, 15, 16, and 17 tempered at 400 F, there were 21 in which the origin was below the surface, as shown in Fig. 4, a view of one fracture surface. It is seen in Fig. 1 that these are the same steels that show the highest fatigue strengths; they all contain 1.3 per cent copper. Specimens of all the other materials showed normal fractures with the crack nucleus on the surface. Likewise, a second heat having approximately the same composition as steel No. 10 was tested (No. 10A), and none of these specimens showed subsurface initiation; also, the fatigue strength was significantly lower than that of the No. 10 steel. This suggests that there were differences in processing of the specimens that raised the surface strength of steels 10, 15, 16, and 17; however, no such difference is known—every effort was made to maintain processing procedures as nearly identical as possible.

It has been previously pointed out that, in general, the steels containing copper as a major alloying element exhibited fatigue properties superior to those having no copper. As shown in Fig. 5 the nominal fatigue strength - yield strength ratio for the copper-bearing steels is larger than that for the other steels. As a possible explanation for this, it is suggested that the fatigue stressing may cause a precipitation of copper. The amount of copper appears to be critical: for the alloys investigated, the optimum fatigue strengths were obtained with a composition containing 1.3 per cent copper.

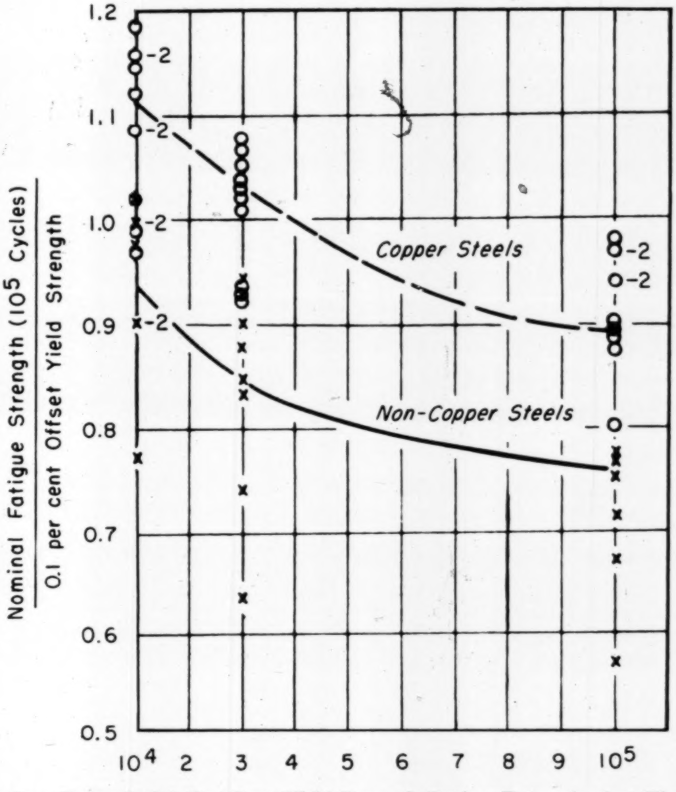


FIG. 5.—Fatigue Strength–0.1 Per Cent Yield Strength Ratios, Determined at Three Points on Each *S-N* Curve.

The number 2 adjacent to a point indicates that the point represents two steels.

CONCLUSIONS

Based on the fatigue strengths at 10^5 cycles of the 18 steels investigated in this program, the following conclusions appear to be warranted:

1. For steels with less than 0.4 per cent silicon, the maximum fatigue strength was generally obtained for the lowest tempering temperature used, either 350 or 400 F. The fatigue strength of high-silicon steels remained approximately constant for tempering temperatures up to 600 F.
2. All of the steels containing 1.3 per cent copper showed higher maximum fatigue strengths than any of the other steels, when tempered at 400 F or lower.
3. For steels of similar alloy content there is a decrease in the fatigue strength-tensile strength ratio with increasing carbon content.
4. Subsurface nucleation of fatigue cracks were prevalent in the steels with the best fatigue properties.
5. The fatigue strength - yield strength ratio is greater for steels containing copper as a major constituent.

With the exception of the points mentioned above, variations in factors other than composition appeared to mask the effects of individual elements on the maximum fatigue strength.

Acknowledgments:

The authors wish to thank Mr. D. F. Armiento, Chief, Thermal Metallurgy Branch of the Pitman-Dunn Laboratories of the Frankford Arsenal for his invaluable assistance and suggestions. Thanks are also due to the Carpenter Steel Co., Crucible Steel Co., and the United States Steel Corp. for some of the steels.

REFERENCES

(1) J. A. Bennett and J. G. Weinberg, "Fatigue Notch Sensitivity of Some Aluminum Alloys," *Journal of Research*, Nat. Bureau Standards, Vol. 52, May, 1954, p. 235 (RP 2495).

(2) G. Sachs, J. D. Lubahn, and L. J. Ebert, "Notched-Bar Tensile Test Characteristics of Heat Treated Low Alloy Steels," *Transactions*, Am. Soc. Metals, Vol. 33, p. 340, (1944).

(3) H. E. Frankel, J. A. Bennett, and W. A. Pennington, "Fatigue Properties of High Strength Steels," *Transactions*, Am. Soc. for Metals, Vol. 52 (1960).

DISCUSSION

Mr. John M. Lessells[1] (*presented in written form*).—In view of the requirements for higher strength steels, the information contained in this paper is of considerable importance. Figure 1 of the paper shows the influence of tempering temperature on the fatigue strength at 10^5 cycles. Since all steels investigated were of extremely high strength, it would have been useful to have included the length of time at the tempering temperature since, as shown by Gillett,[2] this factor may be of extreme importance. To cite one example, an endurance limit of 92,000 psi as obtained from the usual tempering times was increased to 122,000 psi by successive temperings at the same temperature.

This writer believes[3] that this increase may be explained by a decrease in the residual stress, this decrease being brought about by the prolonged times arising from successive temperings. Possibly in future work the authors will take this time factor into account.

Some information regarding the effect of inclusions would be of interest. Cummings et al[4] indicate that the number and type of inclusions may have a bearing on the fatigue strength as well as on the failure location, that is, whether surface or subsurface.

Figure 2 of the paper is of considerable importance and shows the influence of carbon content on the tensile and fatigue properties of these high-strength steels.

Mr. H. E. Frankel (*author*).—With regard to the influence of time at tempering temperature, all of the times were similar; that is, they were 4 hr at the tempering temperatures indicated.

As to the work of Cummings, Stulen, and Schulte[4] regarding the number and type of inclusions and their bearing on fatigue strength as well as on the failure location, whether surface or subsurface, it seems rather surprising, but most, if not all, of the specimens examined had very much the same quantity and distribution of inclusions. These were mainly sulfides and oxides.

Mr. F. B. Stulen.[5]—We are curious as to why most of the failure origins of the specimens were subsurface.

In our work we have found that surface inclusions, or inclusions that were just opened to the surface, were the most detrimental, whereas the fatigue specimens that had the highest fatigue lives were those that had origins located at subsurface inclusions. In our work we stress-relieved all specimens after the final machining operation.

We wonder whether the subsurface origins occurring in the authors' specimens may be indicative of high compressive residual stresses on the surfaces of the specimens, caused by machining, that protected the surface. Did the authors stress-relieve their specimens or otherwise try to eliminate any surface stress condition?

---

[1] Lessells and Associates, Inc., Boston, Mass.

[2] H. L. Gillett and E. L. Mack, "Molybdenum, Cerium and Related Alloy Steels," Chemical Catalog Co., New York, N. Y., p. 202 (1925).

[3] John M. Lessells, "Strength and Resistance of Metals," John Wiley & Sons, Inc., New York, N. Y., p. 173 (1954).

[4] H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Tentative Fatigue Strength Reduction Factors for Silicate-Type Inclusions in High-Strength Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, pp. 505–514 (1958).

[5] Assistant Chief Engineer, Curtiss-Wright Corp., Propeller Division, Caldwell, N. J.

MR. FRANKEL.—No stress-relieving treatments were given the specimens after they were machined. However, with regard to the residual stresses, they are rather elusive things, and I must confess that that was about the only explanation that we could come up with that sounded reasonable with regard to the second heat of one of the materials, in an attempt to duplicate the original results. If you recall, the specimens machined from the duplicate heat did not have the superior fatigue properties exhibited by the original steel, although everything, as far as we know, was identical.

MR. HORACE J. GROVER.[6]—I have two questions.

I was a little bothered that the fatigue strength was evaluated at 10^5 cycles rather than at a longer life, to which I am more accustomed. I noted that even at that life, instead of 3×10^4, one or two materials had fatigue strengths that were at least a high fraction of their yield strength. This would suggest to me the likelihood that the surface stress was above the proportional limit, which I expect to be lower than the yield strength, and I wonder whether some stress values might not be incorrectly given by $\frac{Mc}{I}$.

The second point is this. Eliminating one of the copper-bearing steels and looking at the ratio of fatigue strength to tensile strength, I found it not much larger than that which you would normally expect at this life. Copper seems to have increased the tensile strength and the fatigue strength in about the same ratio.

Connected with that point, I noted that it was some of the copper steels that had some of the subsurface failures. This led me to wonder whether the subsurface failures were due to some unrelieved compressive residual stress. This would be compatible with the apparent effect at only the lower tempering temperatures, since at the higher tempering temperatures there might have been relief of residual stress.

I would appreciate the author's comments.

MR. FRANKEL.—It is quite possible that some of the outer fibers may have been subjected to stresses higher than the proportional limit. However, the stress-strain curve was at such an angle that the 0.1 per cent yield strength was almost identical with the proportional limit. There was a slight difference, but I do not think enough to make much difference, so we were fairly confident that the usual $S = \frac{Mc}{I}$ formula was adequate for the purposes of this investigation.

As for the other points, with regard to the residual stresses and the copper, you certainly have a good point, and I think it merits a great deal of consideration and thought.

MR. H. N. CUMMINGS.[7]—I should like to go back to that question of inclusions Mr. Stulen raised and ask, first, if practically all of the specimens containing copper had subsurface nuclei.

MR. FRANKEL.—No, only the original group of specimens, which contained about 1.28 per cent copper, contained the subsurface nuclei. A second heat to duplicate the original results did not contain them.

MR. CUMMINGS.—Is that dark spot on Fig. 4 of the paper an inclusion?

MR. FRANKEL.—No, but we attempted to knock it out to ascertain what it was.

---

[6] Mechanical Engineering Department, Battelle Memorial Inst., Columbus, Ohio.

[7] Consulting Engineer, Curtiss-Wright Corp., Propeller Division, Caldwell, N. J.

MR. CUMMINGS.—You do not know what was there at the center?

MR. FRANKEL.—No, I do not. We made several attempts on all of the specimens exhibiting this phenomenon but with no success.

MR. CUMMINGS.—I was hoping that we had discovered some way of driving the nuclei subsurface. As Mr. Stulen said, we found that in every case where the nucleus was clearly well below the surface, it took the crack that much longer to work its way up to the surface, and that lengthened the life. So if putting copper in would drive the nuclei subsurface, it might be one way of increasing fatigue life, but I am not so sure now from what you say that that may be a way.

MR. FRANKEL.—No, I would not want to speculate on that. However, as I said, that second heat was a great disappointment.

MR. R. E. PETERSON.[8]—On Fig. 4 of the paper there is a white round spot, with a dark region in the center. Could you suggest an explanation of how this spot is formed?

MR. FRANKEL.—The circle is actually a little below the surface as viewed on Fig. 4, because it is the obverse of the original particle which was on the other part of the specimen. This white spot apparently is the fatigue crack itself, or the result of the fatigue surfaces rubbing together, and apparently there are secondary fatigue cracks coming out from the circumference of the circle.

---

[8] Manager, Mechanics Department, Westinghouse Electric Corp., Pittsburgh, Pa.

LOW-CYCLE FATIGUE OF PRESSURE-VESSEL MATERIALS*†

By G. Sachs,[1] W. W. Gerberich,[2] V. Weiss,[3] and J. V. Latorre[4]

Synopsis

Low-cycle strain-controlled fatigue tests in tension-compression and in bending were conducted on two pressure-vessel materials (A-302 steel and 5454-0 aluminum alloy) and one additional aluminum alloy (2024-T4). It was found that for tension compression cycling the experimental data are well approximated by the equation $N = [(\epsilon_F - \epsilon_0)/\epsilon_{TR}]^2$. In bend cycling the same general trend is observed; however, the slope of the log ϵ versus log N curves is less than that for tension-compression cycling. The effects of mean strain or prestrain, of biaxiality, and of test temperature have been studied. The effects of prestrain or mean strain (cold working) and of biaxiality fade out rapidly and become insignificant somewhere above 10,000 cycles. In contrast, the effects due to differences in ductility of different materials and due to different test temperatures are retained up to at least 100,000 cycles.

List of Symbols

N = number of cycles,
ϵ_p = plastic strain,
ϵ_t = total strain,
ϵ_e = elastic strain,
ϵ_F = natural fracture strain,
ϵ_{TR} = total strain range,
ϵ_{TP} = plastic strain range,
$\epsilon_{\max}$ = maximum strain,
$\epsilon_{\min}$ = minimum strain,
ϵ_0 = mean strain,
R = strain ratio,
σ_1 = principal stress in circumferential direction,
σ_2 = principal stress in transverse direction,
W = width of specimen,
h = thickness of specimen,
k = exponent, constant, and
a = exponent, constant.

---

* This report was prepared by Syracuse University Research Inst. under AEC Contract No. AT(30-1)-2141, with F. W. Davis, Metallurgy Section, Division of Reactor Development, acting as project supervisor, and the Research Committee on Behavior of Pressure Vessel Materials of The American Society of Mechanical Engineers acting as advisory committee.

† Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Professor of Metallurgy, Syracuse University, Syracuse, N. Y.

[2] Project Engineer, Jet Propulsion Laboratory, California Institute of Technology, Pasadena, Calif.

[3] Associate Professor of Metallurgy, Syracuse University, Syracuse, N. Y.

[4] Senior Project Engineer, Syracuse University.

Most of the existing work on fatigue is devoted to large numbers of cycles. In recent years, however, failures after a limited number of cycles have attracted considerable interest. This includes premature failures in aircraft after a few cycles, including fracturing at a low load after a higher load has been carried. Thermal shock or thermal fatigue is now also generally considered a low-cycle fatigue phenomenon.

Experimentation aimed at a basic understanding of low-cycle fatigue is carried out preferably by controlling the strain rather than the stress. High tensile loads, even if controlled closely, lead to large variations in tensile stress and strain because of the shape of the load-extension diagram. This instability is avoided by using strain as the independent variable.

Following the early low-cycle studies by Sachs and his associates between 1948 and 1951 (**1–3**)[5] a large amount of research has been devoted to this problem. The first tests disclosed a gradual slow loss of ductility on strain cycling, and revealed the fact that this fractional reduction in the applied strain decreased as the strain decreased. Manson (**4**) expressed this relation by the equation

$$N^k \epsilon_p = C \qquad (1)$$

where N is the number of cycles to failure, ϵ_p is the plastic component of cyclic strain, and k and C are material constants. This equation has since been applied widely to both mechanical and thermal fatigue (**5**).

In the range of very few cycles the total strain, ϵ_t, can be substituted in Eq 1 for ϵ_p, because the plastic strain is nearly equal to the total strain. The usefulness of this equation for higher numbers of cycles suffers from the fact that the elastic component becomes predominant and the plastic component, therefore, very uncertain. Experimental evidence indicates that within a certain range Eq 1 is a good approximation if $k = \frac{1}{2}$ and $C = \frac{\epsilon_F}{2}$, that is,

$$N^{1/2} \epsilon_p = \frac{\epsilon_F}{2} \qquad (1(a))$$

where the natural fracture strain $\epsilon_F = \ln\left(\frac{A_0}{A}\right)$; A_0 is the original cross section, A the cross section after fracture.

However, it will be shown that the actual value of tensile ductility, ϵ_F, does not yield the best fit and that the exponent k may be considerably lower than one-half. The experimental life, therefore, is frequently higher than that predicted by Eq 1(*a*), and this relation can be used as a safe design criterion.

It will also be shown that the simple equation can be used only for a very limited range of cycles and that, for very low cycles, variables not considered in this relation are of considerable significance.

Thermal fatigue is basically also a strain-cycling fatigue. It differs from mechanical fatigue in that the strains are caused by expansion and contraction of a restrained system and, therefore, progress at a varying temperature. Low-cycle fatigue studies aimed primarily at obtaining design data for thermal fatigue have most recently been conducted on the simpler problem of fatigue by direct strain cycling. The original technique of applying tension and compression by means of a testing machine has also been revived (**5,6**) because of the fundamental significance of the results for a very few cycles.

Fatigue tests are usually performed in bending and direct-load cycling. In strain-cycling investigations direct straining in tension and compression is usually preferred. Strain cycling by bending has been found to be considerably more difficult to control (**7**–**9**), but Low's work (**9**) indicates that substantially the same information is supplied by bending as by tension-compression tests. In contrast, low-cycle stress cycling in bending leads to test data which cannot be correlated with those of direct-stress cycling (**10**).

---

[5] The boldface numbers in parentheses refer to the list of references appended to this paper.

Regarding the relative merits of the lucid and fully controlled direct-strain cycling and the more complicated strain cycling by bending, the following should be considered. Homogeneous straining is very different from the straining caused by heating and cooling, while bending results in a strain and stress distribution similar to that produced by a temperature gradient. Therefore, strain cycling by bending, and thermal fatigue or thermal shock are closely related and permit theoretical and experimental evaluation of more variables than direct-strain cycling.

This paper presents some of the fundamentally significant results of a low-cycle investigation, conducted at Syracuse University. Since the test data will be presented in more detail in a later publication, testing techniques will be discussed here only where necessary.

TABLE I.—DESCRIPTIVE DATA FOR ALLOYS USED.

	2024-T4 Aluminum Alloy	5454-0 Aluminum Alloy	302 Low-Alloy Steel
Form	⅝-in. Plate	⅛-in. Sheet	¾-in. Plate
Condition of Heat Treatment	T-4	0	(*a*) As received (*b*) Normalized and Stress Relieved
Tensile Strength, psi	68 000	36 400 (SURI) 37 000 (Alcoa Research Laboratories)	107 350 104 000
Yield Strength, psi	47 000	13 380 (SURI) 16 000 (Alcoa Research Laboratories)	74 200 68 000
Elongation, per cent	19.0	26.5 (SURI) 20.0 (Alcoa Research Laboratories)	27.00 24.5

MATERIALS AND PROCEDURE

Table I contains pertinent data for the alloys used in this investigation.

Tension-compression cycling tests were made on hourglass-shaped specimens in a universal testing machine (**1**). Bending tests were performed with a machine designed by Prof. A. P. Taber for this investigation, which is similar to that used by Low (**9**) but adapted for work at elevated temperatures.

Strain-cycling tests consist of inducing strains between certain limits. The controlled quantity is usually the total strain, ϵ_{TR}, which consists of an elastic component, ϵ_e, and a plastic component, ϵ_p. The total strain rather than the plastic strain will be considered primarily in this paper. Also, the cycles to failure reported are for complete fracturing. Separate tests were conducted to determine the relation between the cycles to failure and the cycles at which a crack first appeared.

Strains are given here throughout as true or natural strains, that is,

$$\epsilon = \ln\left(1 + \frac{\Delta l}{l}\right).$$

DEFINITIONS IN STRAIN CYCLING

Strain cycling is usually performed between two constant strain values, the maximum strain, $\epsilon_{\max}$, and the minimum strain, $\epsilon_{\min}$. The average value of these, referred to as the mean strain,

$$\epsilon_0 = \tfrac{1}{2}(\epsilon_{\max} + \epsilon_{\min}) \quad \ldots\ldots\ldots (2)$$

has much the same significance as the mean stress in stress cycling, and their sum, the strain range,

$$\epsilon_{TR} = \epsilon_{\max} - \epsilon_{\min} \quad \ldots\ldots\ldots\ldots (3)$$

corresponds to twice the alternating stress in stress cycling.

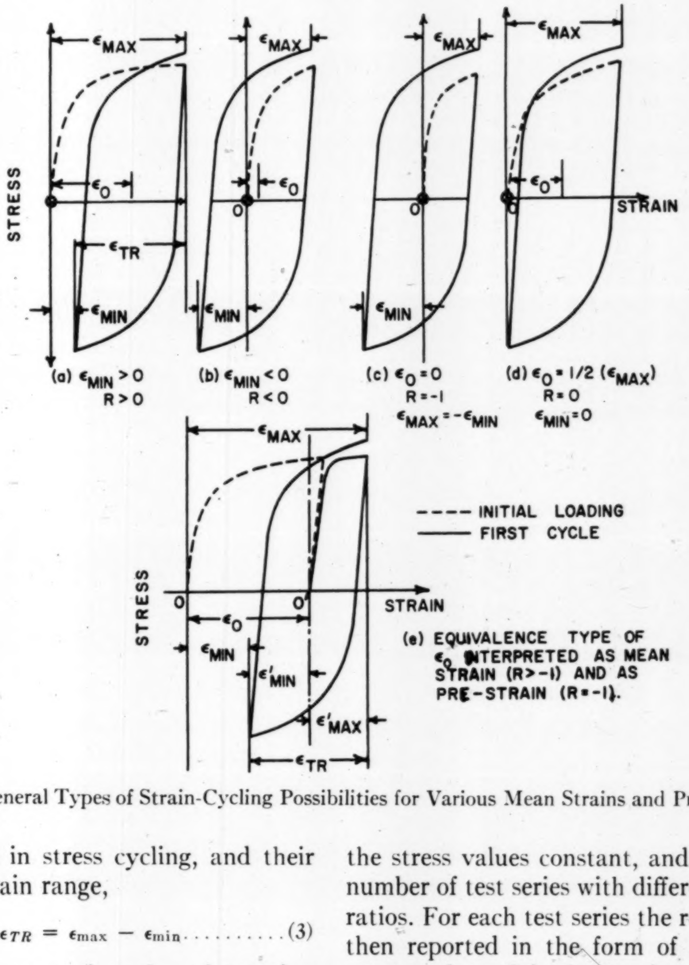

ϵ_{MAX}
STRESS
ϵ_0
ϵ_{TR}
ϵ_{MIN}
0
STRAIN
(a) $\epsilon_{MIN} > 0$
$R > 0$
(b) $\epsilon_{MIN} < 0$
$R < 0$
(c) $\epsilon_0 = 0$
$R = -1$
$\epsilon_{MAX} = -\epsilon_{MIN}$
(d) $\epsilon_0 = 1/2\ (\epsilon_{MAX})$
$R = 0$
$\epsilon_{MIN} = 0$
---- INITIAL LOADING
—— FIRST CYCLE
0'
ϵ'_{MIN}
ϵ'_{MAX}
(e) EQUIVALENCE TYPE OF ϵ_0 INTERPRETED AS MEAN STRAIN ($R > -1$) AND AS PRE-STRAIN ($R = -1$).

FIG. 1.—General Types of Strain-Cycling Possibilities for Various Mean Strains and Prestrains.

The common practice in the past has been to present either the total strain range, ϵ_{TR}, or more frequently, its plastic component, the plastic strain range, ϵ_{TP}, as a function of the number of cycles to failure. As the plastic component is difficult to establish, particularly for large strains, the discussions here are based primarily on the total strain (range).

In order to cover the most important variables it is common practice in stress-cycling work to keep the ratio between the stress values constant, and to run a number of test series with different stress ratios. For each test series the results are then reported in the form of S-N diagrams, where S is the maximum stress. Similarly in strain-cycling work a parameter, or strain ratio,

$$R = \epsilon_{\min}/\epsilon_{\max} = (2\epsilon_0/\epsilon_{\max}) - 1 \quad \ldots (4)$$

can be introduced. The results of different test series can be plotted with $\epsilon_{\max}$ as ordinate, N as abscissae and R as parameter. It is more common practice in strain-cycling studies to report the test data in form of curves which show the strain range, ϵ_{TR}, as a function of the number of cycles, N.

MEAN STRAIN AND PRESTRAIN

The mean strain has not only a formal significance as a strain function, but also a very distinct physical significance. Considering a given value of strain range, the mean strain may vary between certain limits. Several general possibilities exist, two of which are shown in Figs. 1(*a*) and (*b*). Furthermore, two specific cases are of particular significance, $\epsilon_0 = 0$ or $R = -1$, Fig. 1(*c*), and $\epsilon_0 = \frac{1}{2}$ ($\epsilon_{\max}$) or $R = 0$, Fig. 1(*d*). These correspond to reversed and zero-to-maximum stress cycling, respectively.

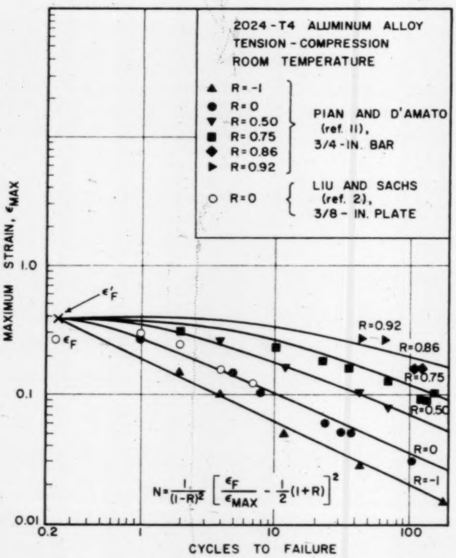


FIG. 2.—$\epsilon_{\max}$ versus N Curves for 2024-T4 Aluminum Alloy in Tension-Compression for Various Strain Ratios, $R = \frac{\epsilon_{\min}}{\epsilon_{\max}}$.

According to Fig. 1(*e*), ϵ_0 can also be interpreted as a prestrain by which the metal had been first strain-hardened and the ductility reduced, if we regard 0′ as the origin of our coordinate system. The subsequent cycling is then performed on this strain-hardened metal always with a mean strain equal to zero. The only difference between the two concepts is the presence of unloading and reloading in prestraining. This should be of no significance, provided that the beginning of strain cycling is in the same direction as the prestrain. (This will be the only case considered here.)

EFFECTS OF PRESTRAIN ON TENSION-COMPRESSION STRAIN CYCLING

Most strain-cycling investigations have been conducted with a strain ratio $R = 0$. Tension-compression tests with different strain ratios or prestrains were conducted to a limited extent at Syracuse University. They were discontinued, since Pian and D'Amato (**11**) reported the results of such work on a material similar to that used by the authors and with corresponding results. The number of cycles in their tests extended from 2 to about 200.

Figure 2 shows the results of their tests. It reveals that for a given value of $\epsilon_{\max}$ the number of cycles to failure greatly increases with increasing R. It is also evident for low values of N that a certain value of $\epsilon_{\max} = \epsilon_F$ cannot be exceeded, where ϵ_F appears to be a value close to the tensile ductility of the material.

The actual value of ϵ_F is rather doubtful, for various reasons. In tension tests on ductile metals necking occurs, and the triaxiality associated with it reduces the ductility from the value to be expected for uniaxial straining (**12**). The test data in Fig. 2 also illustrate that several cycles to failure were obtained with values of maximum strain which exceeded those determined directly.

The more familiar representation of strain-cycling data by means of strain-range–cycle (ϵ_{TR}-N) curves, is given in

Figs. 3 and 4 for the results of the tension-compression tests on 2024-T4 aluminum alloy. Included in Fig. 3 are some earlier data by Liu and Sachs (2) and results of tests at Syracuse University. Figure 3 shows the test data plotted separately, and Fig. 4 assembles the data by Pian and D'Amato in a single graph. It is evident from this graph that the strain ratio, R, has a pronounced effect on the shape and location of the ϵ_{TR}-N curve at small numbers of cycles to failure. Only the data for $R = -1$ appear to follow a straight-line relation, but it terminates at $N = \frac{1}{4}$ in a value greatly in excess of the tensile ductility, ϵ_F. The data for $R = 0$ are considerably better represented by a curved, than by a straight line, and they come closest to terminating in a value near the tensile ductility. The data for strain ratios greater than zero are located considerably below those for $R = 0$ and terminate in correspondingly reduced values at

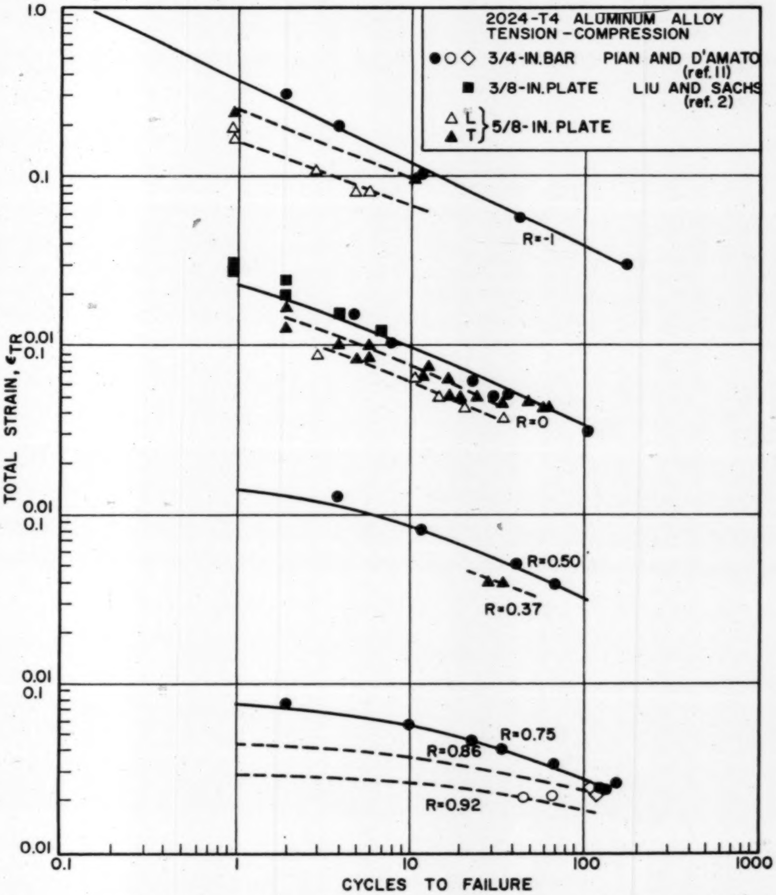


FIG. 3.—ϵ_{TR} versus N Curves for 2024-T4 Aluminum Alloy in Tension-Compression for Various Strain Ratios R.

$N = \frac{1}{4}$. This is to be expected from the concept that the limiting value should be the tensile ductility reduced by the prestrain.

At large numbers of cycles all ϵ_{TR}-N curves in Fig. 4 approach each other to such an extent that their eventual merging should be expected.

The solid lines in Figs. 2 to 4 are drawn according to the concept and equation presented in the next section. This explains certain deviations of the curves from the data points.

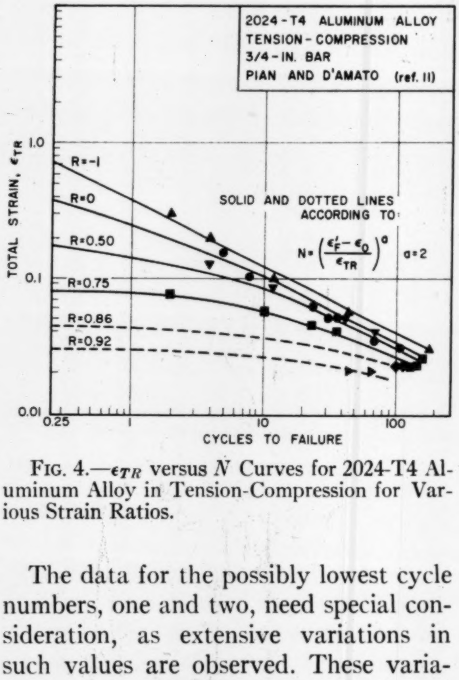


FIG. 4.—ϵ_{TR} versus N Curves for 2024-T4 Aluminum Alloy in Tension-Compression for Various Strain Ratios.

The data for the possibly lowest cycle numbers, one and two, need special consideration, as extensive variations in such values are observed. These variations are explained by the mechanism of low-cycle fatigue, as discussed in Appendix I.

EQUATION OF STATE FOR STRAIN CYCLING IN TENSION-COMPRESSION

It is clear from the test results in Figs. 2 to 4 that no equation that neglects the variable R will be able to represent satisfactorily these data. However, the following equation has been found to yield a good approximation to the experimental data:

$$N = [(\epsilon_F - \epsilon_0)/\epsilon_{TR}]^a \ldots\ldots\ldots (5)$$

The static ductility, ϵ_F, in this equation is not any experimental value but is obtained by curve fitting. The value of the constant a also will not be postulated, but it appears to be always close to 2.

In order to determine these constants, series of tests with $R = -1$ ($\epsilon_0 = 0$) can be used. For this case Eq 5 reduces to

$$N = (\epsilon_F/\epsilon_{TR})^a \ldots\ldots\ldots\ldots (6)$$

or a straight line in log-log coordinates:

$$\log N = a \log (\epsilon_F/\epsilon_{TR}) \ldots\ldots\ldots (7)$$

yielding the constant a from the slope of this line. The value of ϵ_F must be obtained by trial and error, but for $a = 2$ the equation yields, for $N = \frac{1}{4}$

$$\epsilon_F = \epsilon_{\max} = \tfrac{1}{2}\epsilon_{TR} \ldots\ldots\ldots (6(a))$$

Although the exponent a has been found to deviate slightly from 2, it appears that Eq 7 with $a = 2$ is a usable approximation.

If $\epsilon_{\max}$ rather than ϵ_{TR} is used as the ordinate to plot the data, this equation becomes:

$$N = \frac{1}{(1 - R)^2}\left[\frac{\epsilon_F}{\epsilon_{\max}} - \frac{1}{2}(1 + R)\right]^2 \ldots (8)$$

The solid lines in Figs. 2 to 4 are drawn according to this equation, with $\epsilon_F = 0.364$. The lines of $\epsilon_{\max}$ versus ln N become straight lines (Eq 8) for larger numbers of cycles, where $\epsilon_F/\epsilon_{\max} \gg \frac{1 + R}{2}$. In this range the vertical spacing of the curves is a function of R only according to

$$\frac{\epsilon_F^2}{N} = [(1 - R)\epsilon_{\max}]^2 \ldots\ldots\ldots (9)$$

In this analysis of test data the total strain, rather than the plastic strain, is

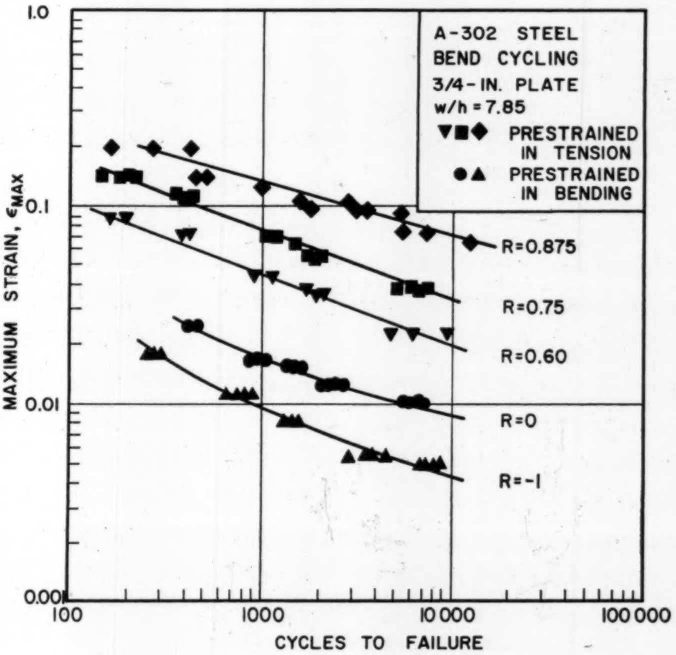
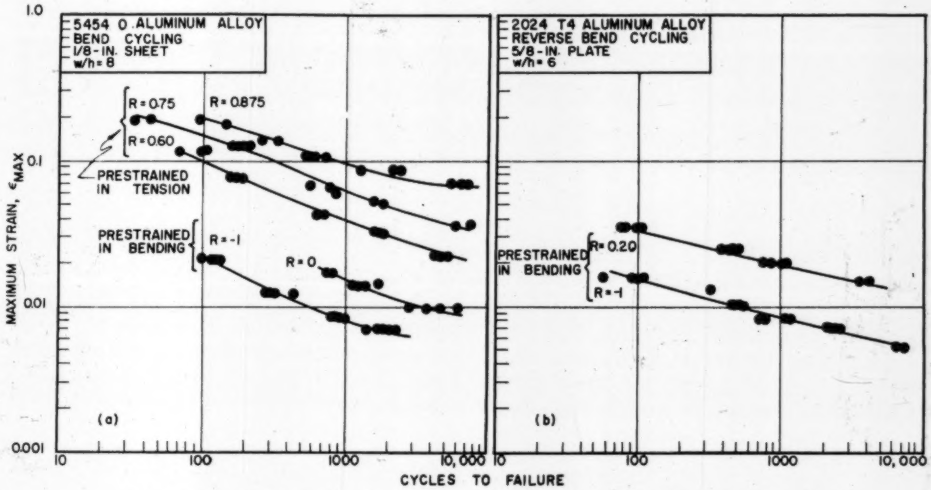


FIG. 5.—$\epsilon_{\max}$ versus N Curves for A-302 Steel in Bend Cycling for Various Strain Ratios.



(*a*) 5454-0 aluminum alloy.

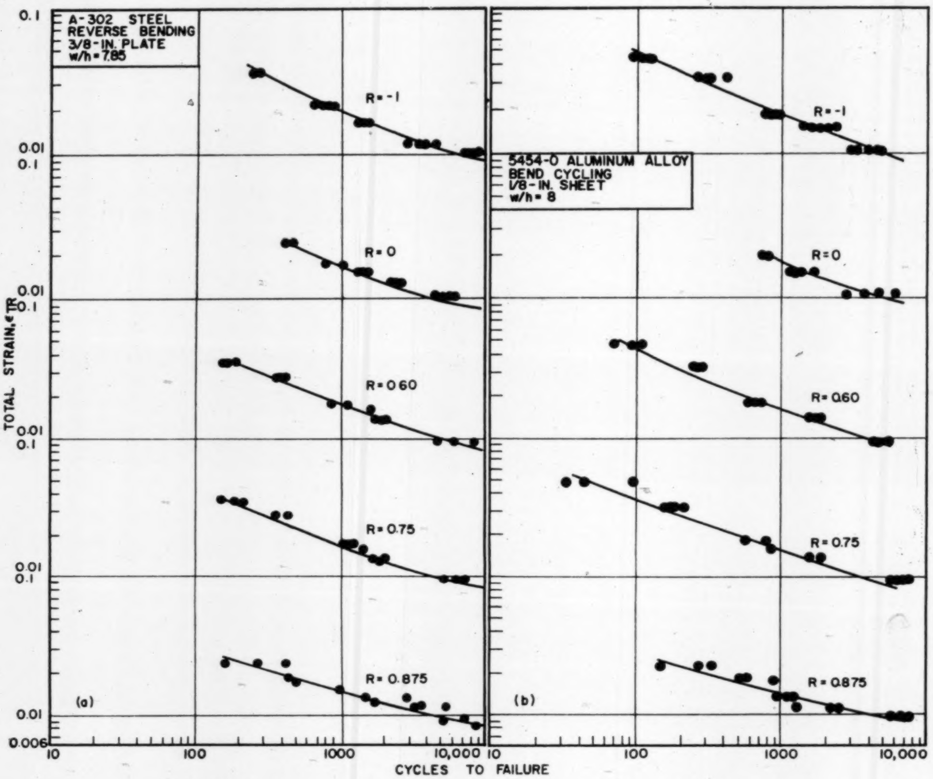
(*b*) 2024-T4 aluminum alloy.

FIG. 6.—$\epsilon_{\max}$ versus N Curves for Aluminum Alloys in Bend Cycling for Various Strain Ratios.

used. For the small number of cycles considered the difference between the two is of little significance. As far as the validity of such an equation for larger numbers of cycles is concerned, it has been found impossible in this investigation to calculate the plastic strains with any degree of reliability, because it is small in comparison with the elastic strains. Therefore, the total strain is used throughout this investigation.

STRAIN CYCLING IN REVERSED BENDING

The results obtained from reversed bending are less lucid than those obtained from tension-compression tests and are at present not amenable to mathematical analysis, compare Figs. 5, 6(*a*) and (*b*), and 7(*a*). There are several well recognized reasons for this complexity of strain cycling in reversed bending, particularly the following:



(*a*) A-302 steel.

(*b*) 5454-0 aluminum alloy.

FIG. 7.—ϵ_{TR} versus N Curves for a Steel and an Aluminum Alloy in Bend Cycling for Various Strain Ratios.

1. Non-homogeneous stress distribution in both the thickness and longitudinal direction, which does not lend itself readily to determination of stresses and, to a lesser degree, of strains.

2. Stress and strain biaxiality, which are known to be functions of the width-to-thickness ratio.

3. Failure to obtain results at low numbers of cycles, that is, high strains because of excessive curvature, and also of buckling.

4. The fact that cracking occurs appreciably before failure, which makes it difficult to define and record the end of the test.

These results, although complicated, are in agreement with the concept that failure in either static tension or static bending occurs at the same maximum stress.

For the stress biaxiality at the center of a surface of a wide sheet subjected to pure bending the theory of plasticity predicts the value $\sigma_2/\sigma_1 = \frac{1}{2}$, where σ_1 is the principal stress in the circumferential direction and σ_2 that in the transverse direction. Strain-gage measurements have shown that a maximum biaxiality close to $\frac{1}{2}$ is obtained whenever the ratio of width, W, to thickness, h, is greater than 8. For values of W/h below 8 the stress biaxiality varies approximately linearly with W/h, see Appendix II.

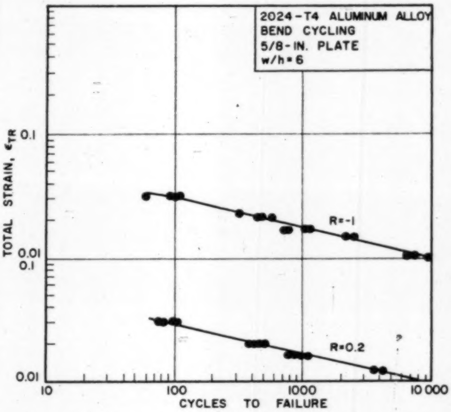


FIG. 8.—ϵ_{TR} versus N Curves for 2024-T4 Aluminum Alloy in Bend Cycling for Various Strain Ratios.

As far as the strains are concerned, failure always occurs at the surface which is subjected to the highest tensile stresses and strains. In bending, furthermore, failure should and does occur in the center of the surface subjected to equal circumferential straining, because biaxiality is present and increases from the edge to the center. It may be mentioned that this mode of bending failure previously observed in static bending also applies to strain cycling.

In this study the maximum attainable total strain range was approximately 0.040. This low strain value leads to a fatigue life above 80 cycles, thus eliminating the very low cycle range for which good agreement with mathematical concepts has been obtained in tension-compression cycling. In addition, in order to obtain strain ratios $R > 0$, the specimens had to be prestrained in tension.

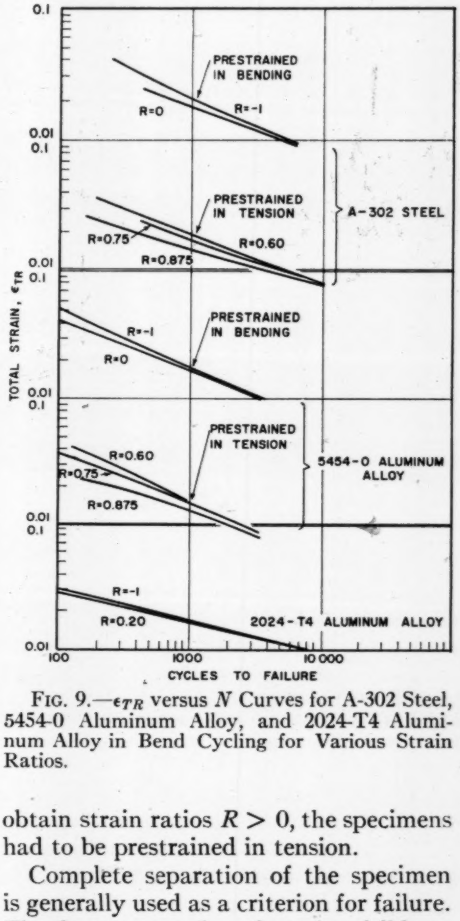


FIG. 9.—ϵ_{TR} versus N Curves for A-302 Steel, 5454-0 Aluminum Alloy, and 2024-T4 Aluminum Alloy in Bend Cycling for Various Strain Ratios.

Complete separation of the specimen is generally used as a criterion for failure. The first appearance of cracks visible to the naked eye occurred before the advent of final fracture, usually at about 80 per cent of the number of cycles to failure, see Appendix III.

In the following the effects of mean strain (strain ratio), biaxiality, and test

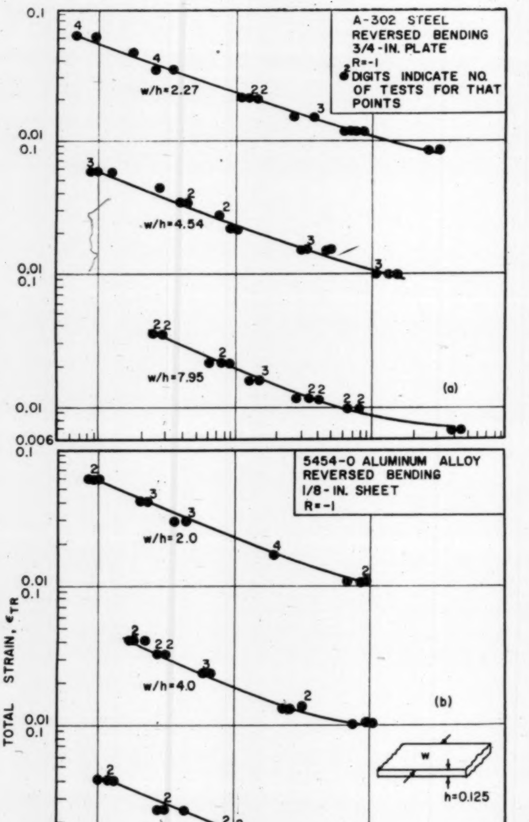


FIG. 10.—ϵ_{TR} versus N Curves for a Steel and Two Aluminum Alloys in Reversed Bending for Various Width-to-Thickness Ratios.

(*a*) for A-302 steel.
(*b*) for 5454-0 aluminum alloy.
(*c*) for 2024-T4 aluminum alloy.

temperature on low-cycle fatigue in reversed bending are discussed for two pressure-vessel materials, a low-alloy steel (A-302) and an aluminum alloy (5454). In addition, a few tests on the aluminum alloy 2024 were conducted, in order to connect the results of bend tests with those of tension-compression tests, and to illustrate the effect of ductility.

EFFECT OF PRESTRAIN ON STRAIN CYCLING IN REVERSED BENDING

The effect of prestrain on reversed-bending, low-cycle fatigue is shown in Figs. 5 to 9 for the widest specimens used in these tests. The number of cycles to failure as a function of $\epsilon_{\max}$, with the strain ratio as a parameter, are plotted in Figs. 5 and 6 for A-302 steel and 5454-0 and 2024-T4 aluminum alloys. The curves show much the same trend as the tension-compression curves at the highest number of cycles (see Fig. 2). They are approximately parallel on a log-log plot, and the curves for higher strain ratios, *R*, are above those for lower strain ratios.

In order to obtain strain ratios greater than zero it was necessary to prestrain the specimens in tension. The vertical separation of the curves for different *R* values agrees with the theoretical predictions for tension-compression cycling (see Eq 9), when the same method of prestraining was used.

Prestraining in tension shifts the curves slightly upward, which causes a somewhat larger than predicted separation between the two curves for prestraining in bending and the three curves for prestraining in tension.

In Figs. 7 and 8 the data are plotted using the total strain range rather than the maximum strain, the strain ratio *R* again being the parameter. In this representation the curves had to be separated in order to avoid overlapping.

Figure 9 is a summary plot of the data presented in Figs. 7 and 8, in which individual data points are omitted for clarity. Furthermore, the curves for prestraining in bending are given separately from those for prestraining in tension, because of overlapping. This means that the curves for prestraining in tension are slightly higher than expected, as compared with the curves for prestraining in bending.

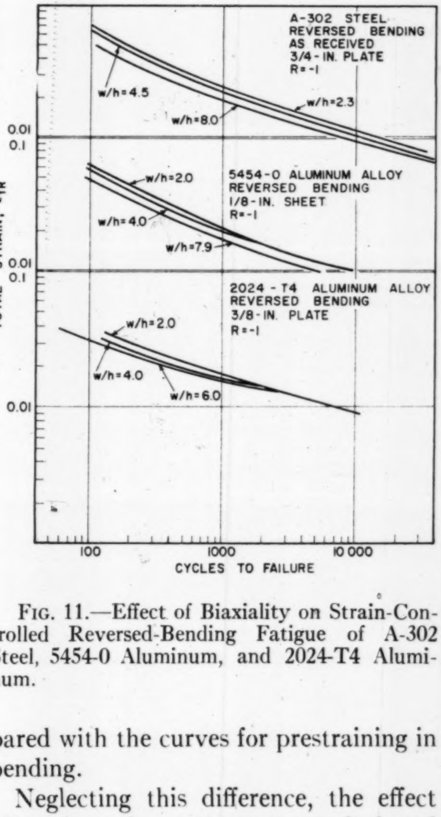


FIG. 11.—Effect of Biaxiality on Strain-Controlled Reversed-Bending Fatigue of A-302 Steel, 5454-0 Aluminum, and 2024-T4 Aluminum.

Neglecting this difference, the effect of strain ratio on these curves is found to be very similar to that derived from tension-compression curves (see Fig. 3). The curves for different strain ratios are well separated at 100 cycles and appear to merge into one curve beyond 10,000 cycles.

EFFECT OF BIAXIALITY

In bend specimens of different width the surface fibers at the center are subjected to a stress biaxiality which increases with increasing ratio of width to thickness, W/h. Therefore, tests were made for each material on specimens having W/h values between 2 and 8 (for a strain ratio $R = -1$). The results of these tests are shown in Fig. 10.

Figure 11 is a summary plot in which the data points are omitted. It can be seen from this graph that the curves are located lower as the biaxiality increases, although the effect is quite small at about 100 cycles and nearly nil at 10,000 cycles.

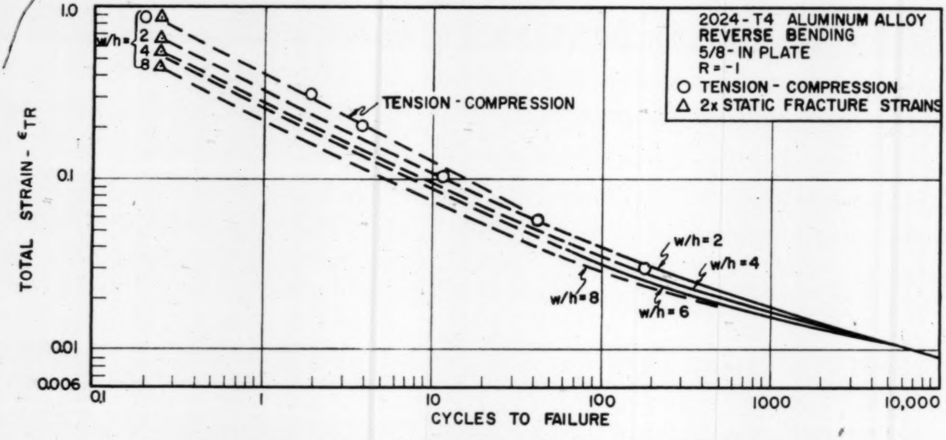


FIG. 12.—Effect of Biaxiality on Strain-Cycling Fatigue Life of 2024-T4 Aluminum.

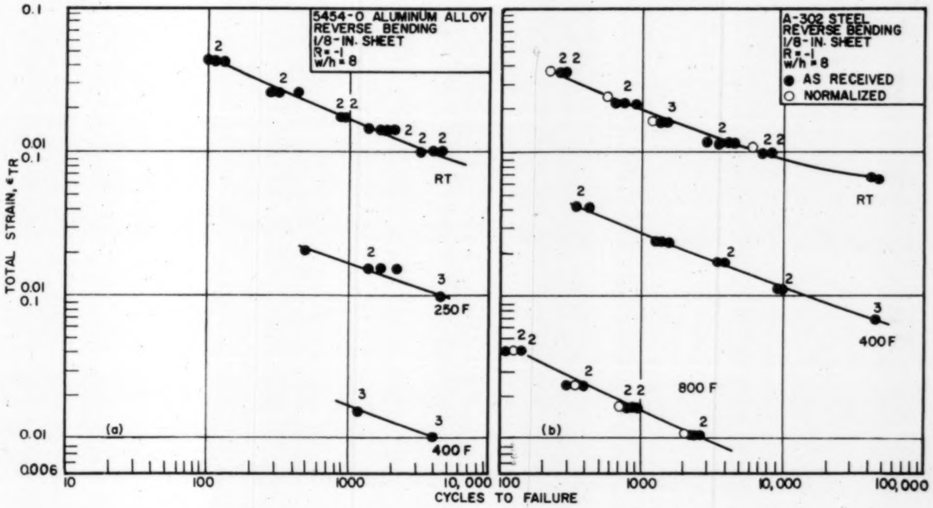
These limited data are also in agreement with the fact that the static value of ductility is considerably reduced by biaxiality, as described in Appendix II. The trend lines in Fig. 11 can be extrapolated to the static ductility values, as illustrated in Fig. 12, indicating that some spread in these curves could be expected at very low numbers of cycles.

EFFECT OF TEMPERATURE

The two pressure-vessel materials, A-302 steel and 5454 aluminum alloy, are subjected to service at elevated temperatures. Completely reversed bend-cycling tests at elevated temperatures were therefore made with these two materials; the results are shown in Fig. 13. Figure 14 is a summary plot of these data in which individual data points are omitted.

The effect of test temperature on 5454-0 aluminum alloy is shown in Fig. 13(*a*). It is not feasible to draw representative curves through the data points available at present, especially for 400 F. The curve for 250 F is found to be nearly parallel to and only slightly above that for room temperature. Further experiments at elevated temperatures are in progress on this alloy.

Steels frequently exhibit reduced ductility within a certain range of test temperatures. The cycling data for A-302 steel in Figs. 13(*b*) and 14 indicate the presence of such an embrittlement range. The strain, at cycles up to 100,000, becomes greater as the test temperature is raised from room temperature to 400 F. However, at 800 F the curve is shifted to values considerably below those at room temperature. It should be noted that these tests were performed at a strain rate of 0.025 in. per in. per sec and that it is known that the embrittlement range in single-load tests varies with the strain rate. Experimentation on the effect of strain rate is in progress.



(*a*) 5454-0 aluminum. (*b*) A-302 steel.

FIG. 13.—ϵ_{TR} versus *N* Curves for a Steel and an Aluminum as a Function of Test Temperature.

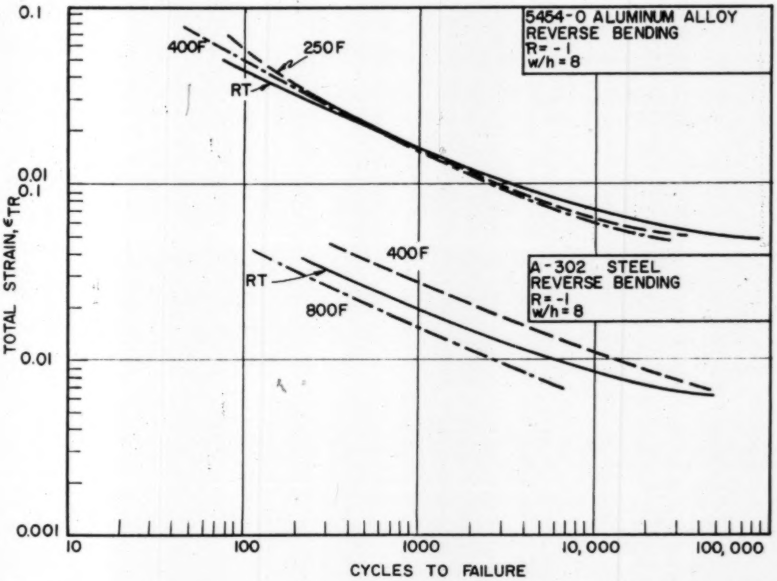


FIG. 14.—Effect of Test Temperature on the Strain-Controlled Reversed-Bending Fatigue Life of 5454 Aluminum and A-302 Steel.

Most of the tests on A-302 steel were conducted with this material in the as-received (hot rolled) condition. A few additional tests were made on specimens normalized at 1650 F and tempered at 1150 F. The results of these tests, included in Fig. 13(*a*), indicate that the two conditions lead to nearly identical results for strain cycling at various temperatures between 100 and 10,000 cycles.

CONCLUSIONS

Considering the strain range that leads to failure at a certain number of cycles in strain-fatigue tests it is shown that a number of variables which greatly affect the ductility in static tests retain this effect in the low-cycle range. These effects are of two types:

The effects of prestraining (cold working) and biaxiality, which are found to fade out rapidly and to become insignificant somewhere above 1000 cycles.

The effects due to differences in the ductility of different materials and due to different test temperatures, in contrast, are retained up to 100,000 cycles.

REFERENCES

(1) S. I. Liu, J. J. Lynch, E. J. Ripling, and G. Sachs, "Low Cycle Fatigue of Aluminum Alloy 24ST in Direct Stress," *Transactions*, Am. Inst. Mining Metallurgical Engrs., Metals Division, p. 469, (1948).

(2) S. I. Liu and G. Sachs, "The Flow and Fracture Characteristics of the Aluminum Alloy 24ST After Alternating Tension and Compression," *Transactions*, Am. Inst. Mining Metallurgical Engrs., Metals Division, p. 193 (1949).

(3) S. I. Liu, "Effects of Precompression on the Behavior of 24ST4 During Cyclic Direct Stressing," *Journal of Metals*, Vol. 3, p. 452 (1951).

(4) S. S. Manson, "Behavior of Materials Under Conditions of Thermal Stress," *Technical Note No. 2933*, Nat. Advisory Committee for Aeronautics (1953); Report No. 1170 (1954).

(5) J. F. Tavernelli and L. F. Coffin, "A Compilation and Interpretation of Cyclic-Strain Fatigue Tests on Metals," *Report No. 57-RL-1847*, General Electric Research Laboratory (1957).

(6) J. H. Gross, S. Tsang, and R. D. Stout, "Factors Affecting Resistance of Pressure Vessel Steels to Repeated Overloading," *Welding Journal* (Research Supplement) Vol. 18, p. 23 (1953).

(7) J. H. Gross, D. E. Gucer and R. D. Stout, "The Plastic Fatigue Strength of Pressure Vessel Steels," *Welding Journal* (Research Supplement) Vol. 19, p. 31 (1954).

(8) J. H. Gross and R. D. Stout, "Plastic Fatigue Behavior of High Strength Pressure Vessel Steels," *Welding Journal* (Research Supplement) Vol. 20, p. 161 (1955).

(9) A. C. Low, "The Bending Fatigue Strength of Aluminum Alloy MG5 Between 10 and 10 Million Cycles," *Journal of the Royal Aeronautical Soc.*, Vol. 59, p. 502 (1955).

(10) G. Sachs and G. Scheven, "Relation Between Direct-Stress and Bending Fatigue of High-Strength Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 667 (1957).

(11) T. H. Pian and R. D'Amato, "Low-Cycle Fatigue of Notched and Unnotched Specimens of 2024 Aluminum Alloy Under Axial Loading," *WADC Technical Note No. 58-27*, Wright Air Development Center, Dayton, Ohio.

(12) G. S. Sangdahl, E. L. Aul, and G. Sachs, "An Investigation of the Stress and Strain States Occurring in Bending Rectangular Bars," *Proceedings*, Soc. Experimental Stress Analysis, Vol. 6, No. 1, p. 1 (1941).

APPENDIX I

FAILURE CONDITION FOR A FEW CYCLES

One of the phenomena unexplained to date is the fact that failure has been observed to occur at a low load after a metal has carried once or repeatedly a considerably higher load. The low-cycle tests explain these observations.

These tests indicate that failure after the first loading—or after a very few cycles—does not occur at a closely defined strain (or stress) but within a range of strains (or stresses). The actual value of the final fracture strain varies with the applied strain range, which may vary within certain limits and still result in failure at the same number of cycles.

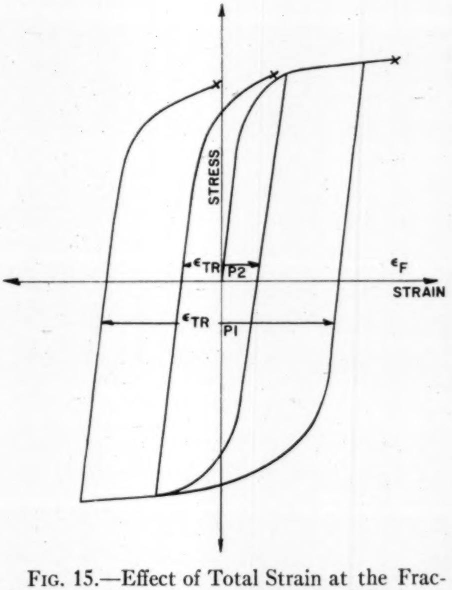


Fig. 15.—Effect of Total Strain at the Fracture Strain for Failure at Nominally 1 Cycle.

In particular, with a strain ratio $R = -1$, failure may occur in the first cycle after a first straining in tension, ranging from close to the static fracture strain in tension down to considerably lower values. The tests by Sachs and Liu (2) have shown that prestraining by a value as low as nearly $\frac{1}{3}$ of the fracture strain will still lead to failure in the first cycle, as illustrated in Fig. 15. The ϵ_{TR}-N curve resulting from these tests is, according to Fig. 16, discontinuous. As the final failure strain in the second tension may now vary from ϵ_F to nearly zero, the number of cycles assigned to these tests may vary from $1\frac{1}{4}$ to $\frac{3}{4}$, according to Fig. 15. This defines, in Fig. 16, a rather steep line within these limits of cycles, or within strains between $2\epsilon_F = 0.76$, and about 0.26. A similar range exists for any larger number of cycles, namely between $N - \frac{1}{4}$ and $N + \frac{1}{4}$, to which all experimental values are limited. Between these ranges no test data are possible, making the ϵ_{TR}-N curve discontinuous, see Fig. 16. For larger number of cycles the discontinuities become very small, but at very low cycles the actual data may deviate considerably from the straight line through the data points terminating at $N = \frac{1}{4}$, $\epsilon_{TR} = 2\epsilon_F$.

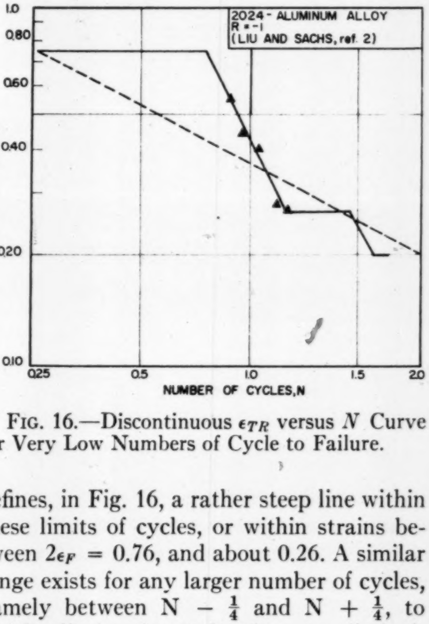


Fig. 16.—Discontinuous ϵ_{TR} versus N Curve for Very Low Numbers of Cycle to Failure.

APPENDIX II

EFFECT OF BIAXIALITY ON FRACTURE STRAIN

When comparing the tension-compression curves for 2024-T4 aluminum alloy in Fig. 12 with the bending curves of a very similar material, one notes that the bending data appear to terminate for small numbers of cycles at a ductility value below that for tension-compression. This difference is caused by the fact that fracturing in tension results from a (nearly) uniaxial stress state, while in a wide bending specimen the failure occurs in the specimen center, where a transverse stress of about one half of the longitudinal stress is present. This biaxiality of 0.5 results from the fact that the transverse strain is then practically zero, or a "plane strain state" exists.

By varying the ratio of width-to-thickness in bend tests various biaxialities can be created.

The effect of the width-to-thickness ratio on the static ductility can be readily determined by means of gridded specimens. The results of these tests are illustrated in Fig. 17, which also includes the results of previous tests by Sangdahl, Aul and Sachs (12) on 2024-T4 aluminum alloy. It can be seen from this graph that the bend ductility considerably decreases as the width-to-thickness ratio increases. It extrapolates to values near the tensile ductility at zero width-to-thickness ratio. For wide specimens, the bend ductility is only approximately one half of the tensile ductility.

These tests will be discussed in more detail in a later report.

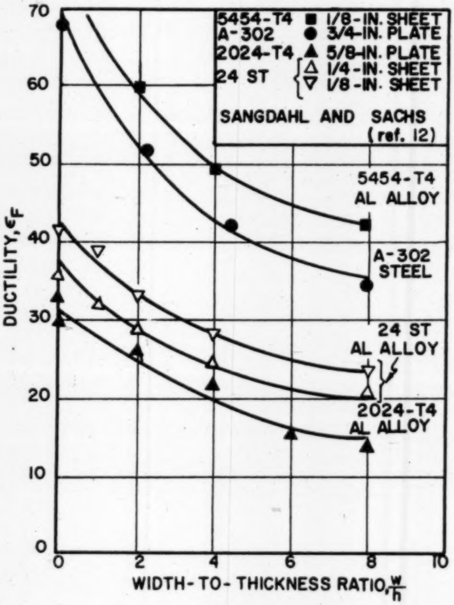


FIG. 17.—Effect of Width-to-Thickness Ratio on Bend Ductility.

APPENDIX III

CRACKING AND FAILURE IN BEND CYCLING

In the main body of this report the effects of all variables and parameters have been referred to the total number of cycles withstood to failure. This failure is preceded by the formation and propagation of cracks.

In order to study the relationship between strain range, number of cycles at which first cracks are observed, and final failure, a number of completely reversed bend tests were conducted. In these tests the first appearance of the crack which led to final failure could be observed with reasonable accuracy. In general, cracking occurred at about 0.8 of the number of cycles to failure, as shown in Fig. 18.

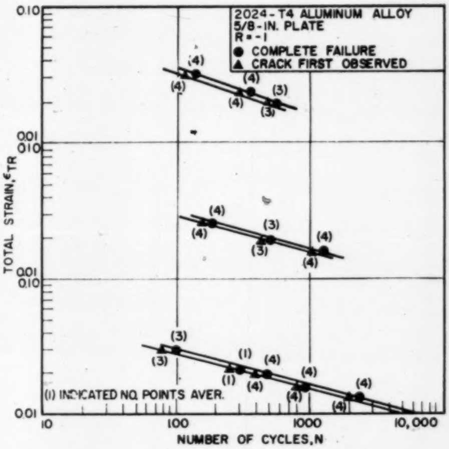


FIG. 18.—ϵ_{TR} versus N Curves for Strain-Controlled Bend Cycling of 2024-T4 Aluminum Indicating First Appearance of Cracks.

ELEVATED-TEMPERATURE TENSILE AND FATIGUE BEHAVIOR OF UNALLOYED ARC-CAST MOLYBDENUM*

By G. W. Brock[1] and G. M. Sinclair[1]

Synopsis

Tension and fatigue tests were carried out over a range of temperatures to explore the possible effects of interaction between interstitial atoms and dislocations on fatigue behavior of recrystallized molybdenum containing small amounts of interstitial elements.

No increase in tensile strength corresponding to that found in the "blue brittle" range in steel was observed as the test temperatures were raised over the range -300 to 1800 F. Values of the upper and lower yield point remain essentially unchanged over the temperature range 200 to 400 F. The presence of a "knee" in the fatigue S-log N diagram over the temperature range 75 to 875 F suggests that strain aging may be influencing the fatigue behavior in this temperature range and may also be responsible for the high values of fatigue ratio (0.65–0.72) observed at these temperatures.

The phenomenon of strain aging in fatigue of steels has been discussed in recent papers. It has been shown (**1**)[2] that ferrous materials capable of strain aging may readily be "coaxed" (that is, run for large number of cycles at several successively increasing values of stress) to a much higher fatigue strength than they formerly possessed. The existence of a sharp knee in the S-N diagram for steel (**2,3**) as well as the increased fatigue life and fatigue limit at slightly elevated temperatures also appear to be intimately related to the phenomenon of strain aging. It has been postulated (**4**) that conditions of stress frequency and temperature for optimum fatigue performance of a strain-aging alloy may be predicted quantitatively from concepts of dislocation–interstitial-atom interaction. Reasonably good agreement has been found (**4**) between the optimum elevated-temperature fatigue behavior of AISI 1018 steel and calculations based on a modification of Nabarro's relation for strain aging (**5**).

Beneficial increases in fatigue strength through strain aging are of possible practical value, especially in aircraft structural materials, and it is believed that a study of strain aging in fatigue should be further advanced to cover nonferrous materials.

Since evidence of strain aging has been reported in studies of the tensile behavior of molybdenum (**6,7,8**), this material was selected for the current investigation.

Purpose and Scope of Investigation

The purpose of this investigation was to explore the behavior of dilute interstitial alloys of molybdenum using tension and fatigue tests at low and elevated temperatures, and to determine the effects (if any) of interaction between interstitials and dislocations on both tensile properties and fatigue limit.

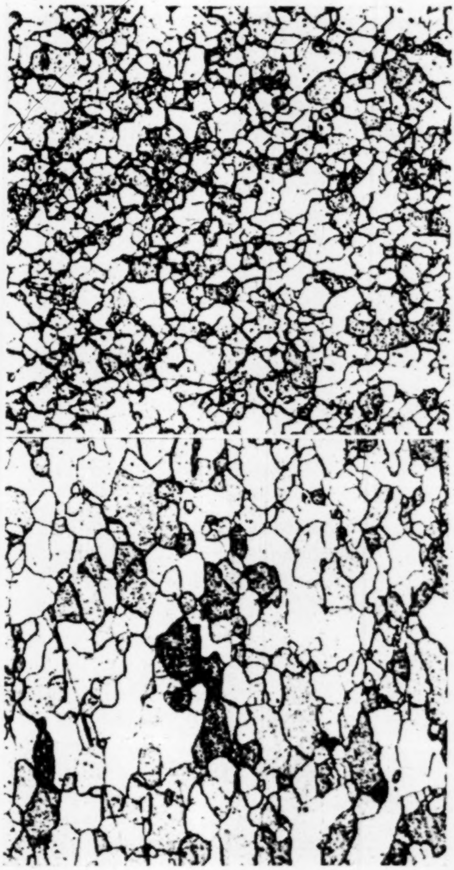
---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Research Associate and Research Professor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

(a) Transverse section.

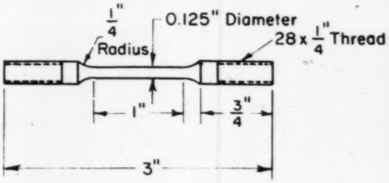


(b) Longitudinal section.

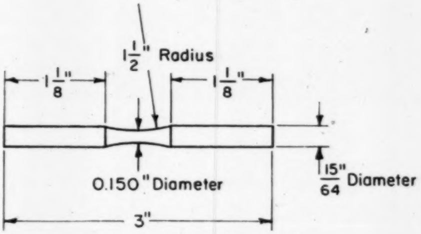
Fig. 1.—Molybdenum Recrystallized at 2800 F for 20 min (× 100).

Accordingly, specimens of commercially pure arc-cast molybdenum were tested in tension throughout the temperature range −300 to 1800 F to determine the effects of strain aging on tensile properties. Rotating cantilever fatigue tests were made at room temperature, 475, 675, 875, and 1100 F with particular emphasis on the fatigue limit to determine what effects strain aging may have on the fatigue behavior.

All tests above 900 F were carried out in an argon atmosphere.



(a) Tension specimen.



(b) Fatigue specimen.

Fig. 2.—Details of Fatigue and Tension Specimens.

Material, Heat Treatment, and Preparation of Specimens

Commercially pure arc-cast molybdenum was obtained from the Westinghouse Electric Corp. in the form of $\frac{1}{4}$-in. diameter swaged bar. A recrystallizing treatment was carried out on this material with a high-temperature, high-vacuum furnace using finish-polished specimens so that any work hardening due to polishing was eliminated. The recrystallizing treatment was carried out at 2800 F for 20 min in a vacuum of less than 0.5 μ. The final grain size averaged about 800 grain per sq mm (ASTM Nos. 6 to 7)[3] on a transverse cross-section and

---

[3] Tentative Methods for Estimating the Average Grain Size of Metals (E 112 – 58 T), 1958 Book of ASTM Standards, Part 3, p. 506.

ASTM Nos. 3 to 6 on a longitudinal cross-section; a photomicrograph of the grain structure is shown in Fig. 1.

The chemical analysis of the material impurities was as follows, values being in weight per cent:

	Carbon	Nitrogen	Oxygen
As-received material	0.0008	0.0023	0.0071
Recrystallized material	0.0054	0.0012	0.0079

The spectrographic analysis of the recrystallized material is given below, values being in parts per million:

Iron	<100
Boron	<50
Cobalt	<100
Cadmium	<100
Manganese	<25
Aluminum	50
Magnesium	<10
Zinc	<100
Tin	<50
Copper	<25
Lead	10
Chromium	100
Silicon	100
Titanium	<25
Nickel	<100
Vanadium	<50
Silver	<20
Sodium	<100
Calcium	<1000
Balance	Molybdenum

Fatigue specimens were machined to the dimensions shown in Fig. 2(*b*) and mechanically polished using successively finer grades of emery paper. The final polish was given with a 00 grade of emery paper and was done in such a manner as to leave the scratches parallel to the axis of the specimen.

METHOD OF TESTING

Tension tests were carried out using an Instron testing machine. This machine autographically records load and deformation and imposes a constant rate of head motion.

The type of specimen used is illustrated in Fig. 2(*a*), the nominal diameter of the specimen being 0.125 in. and the nominal gage length 1.0 in. Head motion of the testing machine was taken as a measure of the specimen elongation.

The mean fatigue limit in rotating bending at a cyclic frequency of 3450 cpm was determined using the staircase statistical method of testing (9) 10 million cycles being defined as a runout. The 95 per cent confidence limits on the mean were also determined from the data.

The fatigue tests up to 875 F were carried out in air, but those at 1100 F were carried out in an argon atmosphere, since oxidation of molybdenum is rapid at this temperature. At 1100 F specimen failures were still being obtained at 25 million cycles, and it was decided to dispense with the staircase method of testing at this temperature to reduce consumption of argon.

Nominal stress at the minimum section of the specimens was computed by means of the ordinary flexure formula:

$$S = Mc/I$$

where:

S = the stress at the outer fiber, psi,
M = the bending moment applied, in.-lb, and
I/c = the section modulus at the critical section, cu in.

RESULTS AND DISCUSSION

Tension Test Results:

Tension tests were carried out at a strain rate of 0.02 in. per in. per min on recrystallized molybdenum to explore the strain-aging properties of the material. The location of a critical temperature region, signified by any increase in strength as the temperature was raised, could then be used to estimate the temperature range of interest in fatigue.

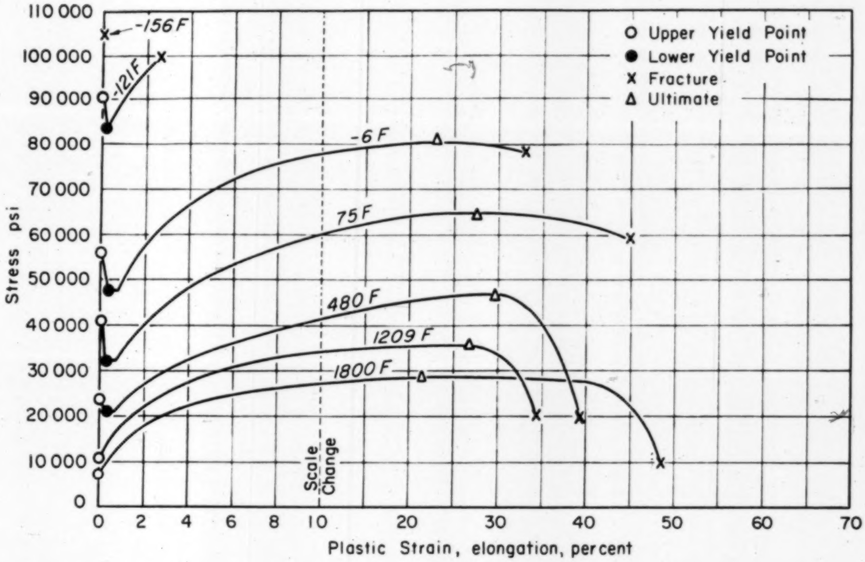


FIG. 3.—Tensile Stress Strain Curves at Various Temperatures for Molybdenum Recrystallized at 2800 F for 20 min.

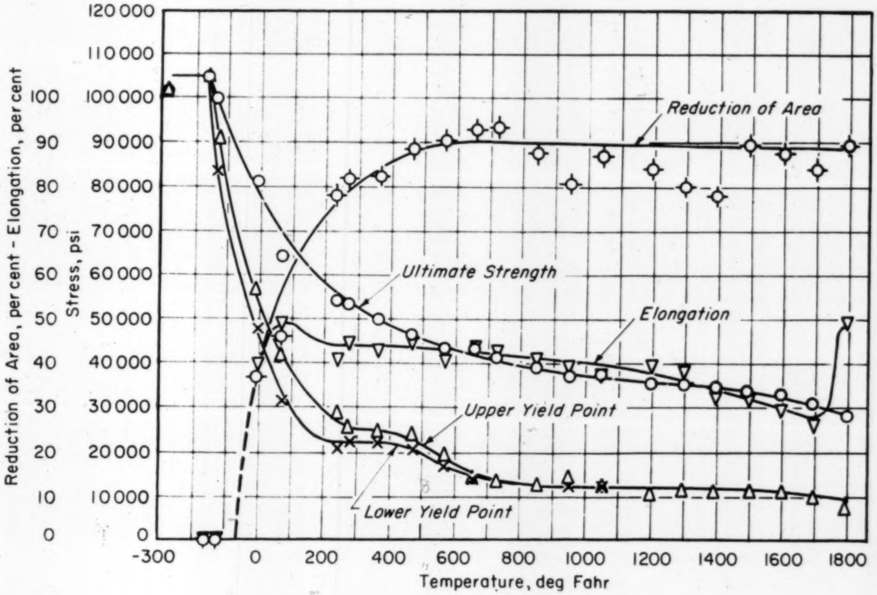


FIG. 4.—Tensile Data for Recrystallized Molybdenum in the Temperature Range −300 to 1800 F.

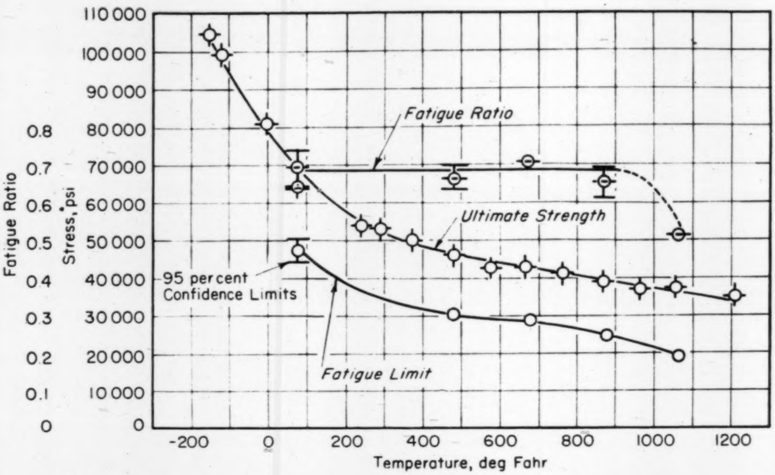


FIG. 5.—Ultimate Tensile Strength, Fatigue Limit, and Fatigue Ratio as a Function of Temperature.

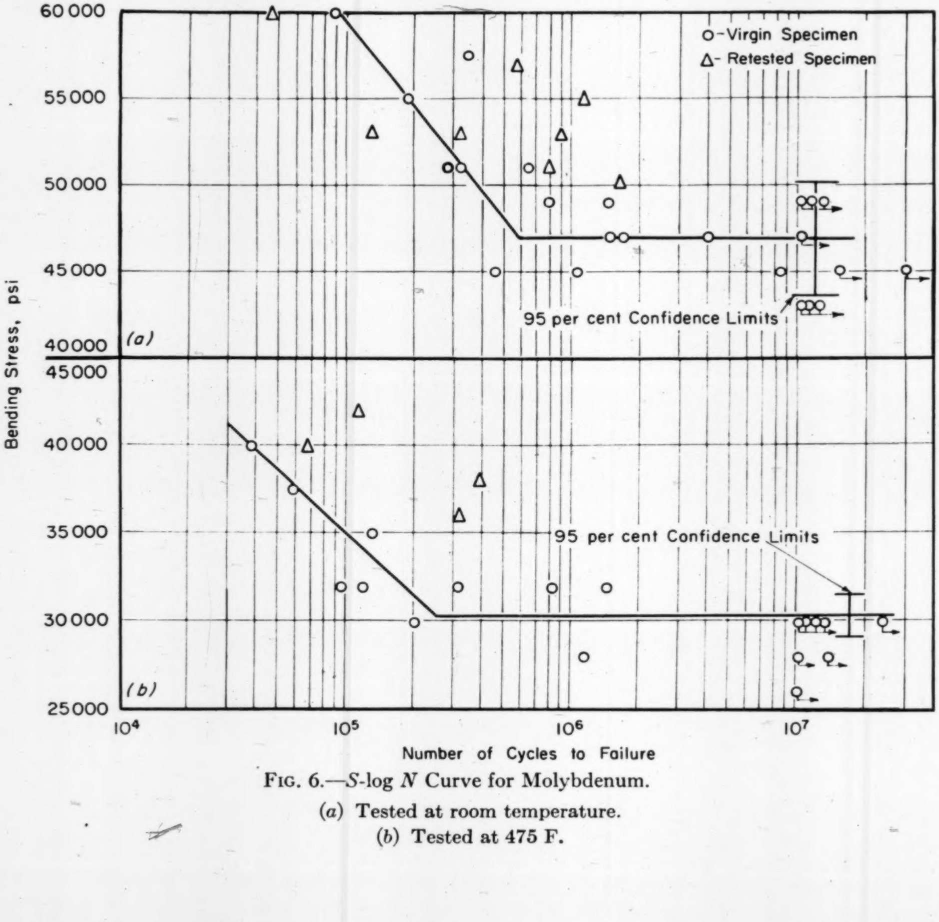


FIG. 6.—*S*-log *N* Curve for Molybdenum.

(*a*) Tested at room temperature.

(*b*) Tested at 475 F.

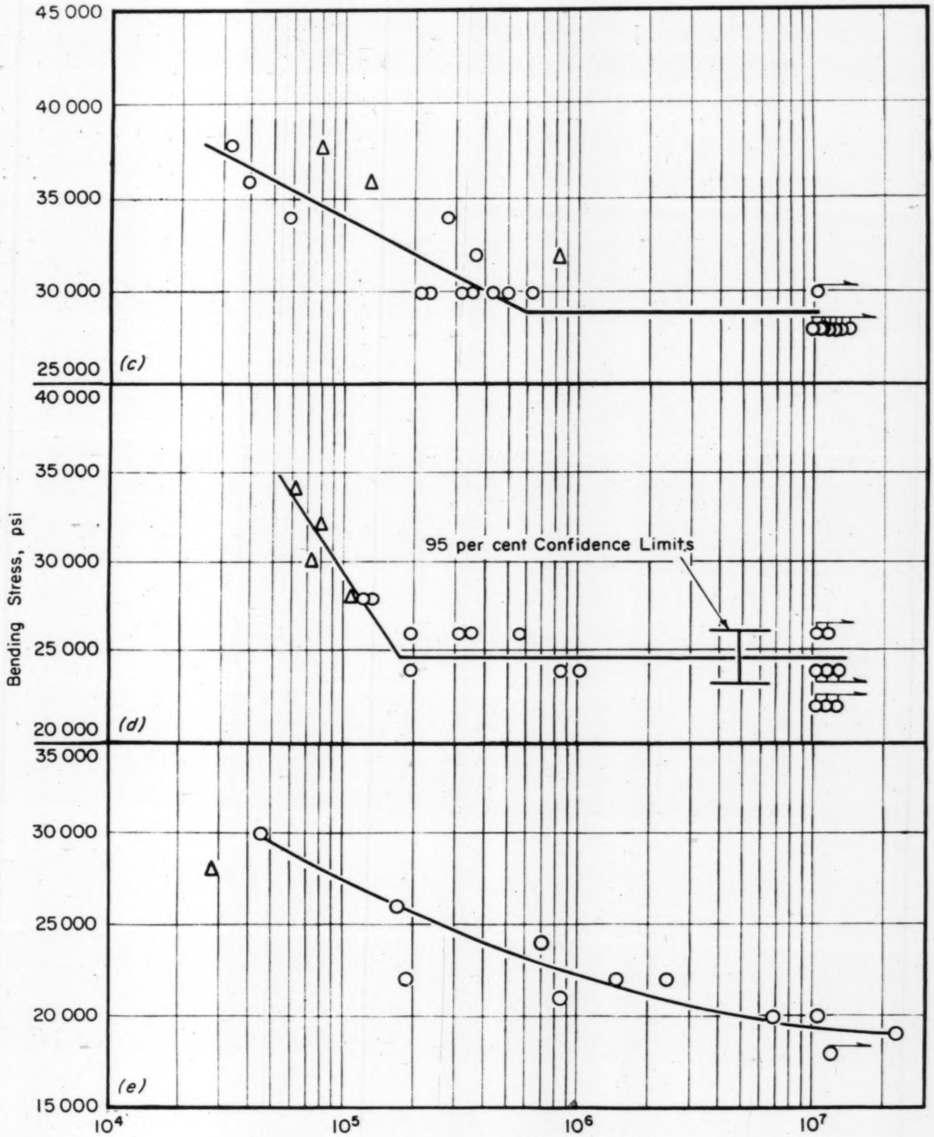


FIG. 6 (*Continued*)
(*c*) Tested at 675 F.
(*d*) Tested at 875 F.
(*e*) Tested at 1100 F.

The temperature range covered in the tension tests was from −300 to 1800 F, and at 900 F and above an argon atmosphere was used to protect the specimens. The type of stress-strain curves obtained at these temperatures is illustrated in Fig. 3, and the tensile properties are shown as a function of temperature in Fig. 4.

Figures 3 and 4 show that the material possessed a pronounced upper and lower yield point in the temperature range −170 to 650 F, but there were traces of an upper yield point up to 1700 F.

The ultimate strength of the material falls rapidly from −170 to 400 F and then shows a much more gradual decrease.

The yield point, like the ultimate strength, falls rapidly at first with increasing temperature but levels off between 200 and 400 F, drops between 400 and 700 F, and then becomes fairly constant up to 1800 F.

At 1800 F there is a sudden increase in the per cent elongation (also reported by Pugh (7)). Between 1400 and 1600 F it was noticed that the flow curve was very serrated, similar to the blue brittle range in steel. Pugh (7) also noted this effect between 1000 and 2000 F.

Discussion of Fatigue Results:

It is apparent from Fig. 5 that in the range from room temperature to 1100 F, the fatigue limit of the material drops steadily with the ultimate strength.

It is interesting to compare the fatigue ratios at each test temperature; this has been done in Fig. 5. Apparently there is no significant difference in the fatigue ratio from room temperature to 875 F, but there is a considerable drop between 875 and 1100 F.

Other differences in fatigue behavior at 1100 F may be observed from Figs. 6(*a*) to (*e*). Apparently the slope of the finite-life portion of the fatigue curves at 875 F and below is such as to give the impression that a knee occurs in the *S*-log *N* diagram, whereas at 1100 F failures are still obtained at 25 million cycles, and it seems doubtful that there is a knee in the *S*-log *N* curve at this temperature. Also, it may be noted that at room temperature, 475, and 675 F, specimens that ran out to 10 million cycles or more and which were then retested at higher stresses have, in most cases, a longer life to failure than virgin specimens tested at the same stress level.

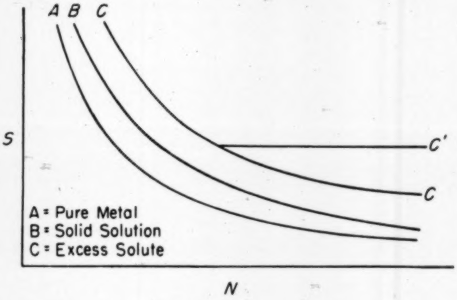


FIG. 7.—Schematic Fatigue Diagram. (After Lipsitt and Horne (3)).

Of the two types of phenomena noted above, namely, (*a*) the presence of a knee in the *S*-log *N* curve and (*b*) increase in the finite life of specimens previously stressed near the fatigue limit, it is interesting to note that both kinds of behavior have been associated with the presence of interstitial atoms, causing strain aging in the material.

Lipsitt and Horne (3), working with iron having very low impurity content, have likened the *S*-*N* fatigue curve of high-purity iron to that of non-ferrous metals (that is, a smooth function of the applied stress). The addition of carbon or nitrogen causes solid-solution strengthening which shifts the *S*-*N* curve to the right and in addition confers the ability to strain age, which further shifts the *S-N* curve upward and to the right (see Fig. 7). Independent of the strengthening due to strain hardening, there would be some limiting lower stress at which the strengthening due to strain aging will be great enough to offset the weakening due to the deformation; this stress, shown by curve *C′*, Fig. 7, is the fatigue limit.

It is possible to use the concept described above to account for the lower fatigue ratio of the molybdenum in fatigue at 1100 F in terms of strain aging.

The fatigue behavior of molybdenum between 75 and 875 F may be represented by curve *C′*, Fig. 7, so that there is the effect of pinning of dislocations giving rise to a higher fatigue strength. However, at 1100 F the efficiency of interstitial atoms in pinning dislocations has been reduced, and the fatigue strength is now more representative of curve *C*, Fig. 7. It is thought that the decreased efficiency of strain aging or pinning at 1100 F is responsible for the lower fatigue ratio at this temperature.

In the temperature range explored, strain aging effects during elevated temperature fatigue and tension testing were found to be rather weak as compared with the behavior of mild steel in the "blue brittle" temperature range.

It is believed that the solid solubility of interstitial elements in molybdenum is too low to enable significant strengthening due to strain aging to occur.

SUMMARY OF RESULTS

1. There is no evidence of increase in tensile strength of the molybdenum with temperature due to strain aging over the temperature range (−300 to 1800 F) investigated.
2. There is a sudden increase in percentage elongation between 1700 and 1800 F.
3. The fatigue ratio (fatigue limit:ultimate strength) remains fairly constant between 0.65 and 0.72 over the temperature range from 75 to 875 F.
4. There is evidence of a knee in the *S*-log *N* fatigue curve over the temperature range 75 to 875 F.

Acknowledgment:

This report was prepared by the Department of Theoretical and Applied Mechanics under USAF Contract No. AF 33(616)-5153. The research was administered under the direction of the Aeronautical Research Laboratory, Directorate of Laboratories, Wright Air Development Center, with Harry A. Lipsitt as cognizant scientist.

REFERENCES

(1) G. M. Sinclair, "An Investigation of the Coaxing Effect in Fatigue of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 759 (1952).

(2) F. C. Rally and G. M. Sinclair, "Influence of Strain Aging on the Shape of the *S-N* Diagram," *TAM Report 87*, University of Illinois, Urbana, Ill. (1955).

(3) H. A. Lipsitt and G. T. Horne, "The Fatigue Behavior of Decarburized Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 586 (1957).

(4) J. C. Levy and G. M. Sinclair, "An Investigation of Strain Aging in Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 866 (1955).

(5) F. R. N. Nabarro, Report on a Conference on Strength of Solids, The Physical Society, p. 38 (1948).

(6) J. H. Bechtold, "Effects of Temperature on Flow and Fracture Characteristics of Molybdenum," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., *Journal of Metals*, Nov. 1953, p. 1469.

(7) J. W. Pugh, "The Tensile Properties of Molybdenum at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 47, p. 984 (1955).

(8) R. P. Carreker and R. W. Guard, "Tensile Deformation of Molybdenum as a Function of Temperature and Strain Rate," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., *Journal of Metals*, Feb. 1956, p. 179.

(9) "A Guide to Statistical Methods for Use in Fatigue Testing," Manual on Fatigue, Am. Soc. Testing Mats. (Committee E-9) on Fatigue) (1958). (Issued as separate publication *ASTM STP NO. 91-A*.)

DISCUSSION

MR. H. J. GROVER.[1]—Were any experiments made on the effect of strain rate in your tension tests, particularly at the lower temperature?

MR. G. W. BROCK (*author*).—No experiments were carried out on the influence of strain rate on the tensile properties. However, experiments on the strain rate and temperature dependence of the ductile to brittle transition in molybdenum subjected to torsional loading have been carried out by my coauthor, Mr. Sinclair, on a different heat of molybdenum. The results of the work are in T and A. M. Report No. 156, University of Illinois, Urbana, 1959.

MR. FRED W. DEMONEY.[2]—Was any cracking observed on the tension specimens at elevated temperatures between 1600 to 2000 F?

MR. BROCK.—No cracks were observed on the tension specimens at any test temperature. At the elevated temperatures the specimens necked down to rather sharp points.

MR. S. E. BRAMER.[3]—Does the author know anything about the process history of the material that he tested?

The reason for my asking this question is that there is a lot of data being generated for refractory metals but a good deal of these data are virtually useless unless we know the process history in making the material. At the present time Task Group AM-4 of the Aircraft and Missile Structural Materials Panel is recommending that standard tests including the processing history of the heat of the material being tested be reported so that a correlation can be attained between process history and property data. If it is possible to have information on the process history of the material from the tests that were run, it would aid us in our work on Task Group AM-4 for it is probably the only work in this country that does have this type of information from all of the refractory metal data that have been generated.

MR. BROCK.—The metallurgical history of $\frac{1}{4}$-in. round rod, prior to its delivery at the University of Illinois was as follows:

The material in the form of $\frac{5}{8}$-in. diameter arc-cast rod was swaged to 0.550 in. and 0.447 in. at 1200 to 1300 C in air, to 0.309 in. at 1150 to 1250 C in air, and then to 0.272, 0.265, and 0.257 in. at 1100 to 1200 C in air. It was then centerless ground to finished size, cleaned in fused potassium hydroxide for 10 to 15 sec, rinsed in hot water, and finally received a diluted hydrochloride rinse in hot water, after which it was wiped dry.

Concerning the effect of its processing history, the most important factor appears to me to be whether it is arc-cast or sintered. Other than that I feel that its final properties will depend mainly on the recrystallizing treatment it receives (that is, temperature of recrystallization, atmosphere used, or the degree of vacuum) and its chemical composition, particularly with regard to its content of the common interstitial elements.

---

[1] Mechanical Engineering Department, Battelle Memorial Inst., Columbus, Ohio.

[2] Head, Mechanical Metallurgy and Evaluation Branch, Kaiser Aluminum and Chemical Corp., Spokane, Wash.

[3] Head, Fabrication Development Section, Hughes Tool Co., Air Division, Culver City, Calif.

Regarding other work published on the properties of refractory metals, I feel that information could be obtained as to the processing history by writing to the authors in question.

NEW OBSERVATIONS RELATING TO THE MECHANISM OF FATIGUE FAILURE*

By Melvin A. Wilkov[1]

In recent years a great deal of attention has been given to the study of the structural changes that occur in a crystalline material under the action of a cyclic stress. The plastic deformation that occurs on a crystal surface due to an alternating stress has been well documented (**1**,**2**,**3**)[2] by several investigators. These deformations have also been compared with the deformation that occurs during a monotonically increasing stress. Investigations have also been conducted using such methods as X-ray diffraction (**4**,**5**) and etch pit techniques (**6**) to obtain a glimpse into the mechanism that causes fatigue failure. Agreement has been reached on the idea that for alternating stress, the nonhomogeneous deformation that takes place can be described by discrete flow units called dislocations and that the forward and reverse deformations do not always take place on the same crystallographic plane. A large amount of local lattice distortion is present in the crystal, some of it being recoverable upon reversing the stress direction. Still the most elusive problem of all is the formation of the microcrack, the actual rupture of the lattice bonds that leads to the formation of a crack. Deformation itself, by a simple slip process, should cause no major structural changes, since only a translation of lattice position occurs. Some other mechanism, other than deformation, must be the controlling process.

The formation of the first phase of fatigue damage, or the formation of an incipient crack, appears to take place on the crystal surface (**7**). Electropolishing away a small surface layer after fatigue cycling seems to cause the material to revert to its original condition. This would indicate that an investigation into the changes that occur on the crystal surface during the application of a fatigue stress would be a promising approach to the understanding of the fatigue process. Previous investigations (**8**,**9**,**10**) in this phase, however, have been limited by the resolution of the microscope or distorted by the means used to amplify the subtle change occurring during the conduct of the test. With the development of the shadowed carbon replica for the electron microscope a means has been provided which is capable of resolution on the order of 20 Å. With this increase in resolution, a further study of the phenomena which constitute initial fatigue damage was thought desirable.

It was the purpose of this study to use the electron microscope in combination with a negative carbon-platinum replica and a means for selected-area examination to extend our knowledge of the basic process leading to the formation of a microcrack.

Material Studied and Method of Testing

The material used throughout this study was SAE 4340 aircraft-quality steel. This is a commonly used engineering material available in a form that is reasonably free of foreign impurities and inclusions. The specimens were prepared with as strain-free a condition as possible by annealing after the machining operations. Annealing was carried out to give a slightly exaggerated grain size in order to facilitate area identification for the replication process. The surface effects developed during annealing were removed by using fine metallographic polishing papers followed by electropolishing in an acetic acid-perchloric acid electrolyte. No surface etching was necessary, as the specimens were slightly etched by the electropolishing operation.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] The Pennsylvania State University, University Park, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

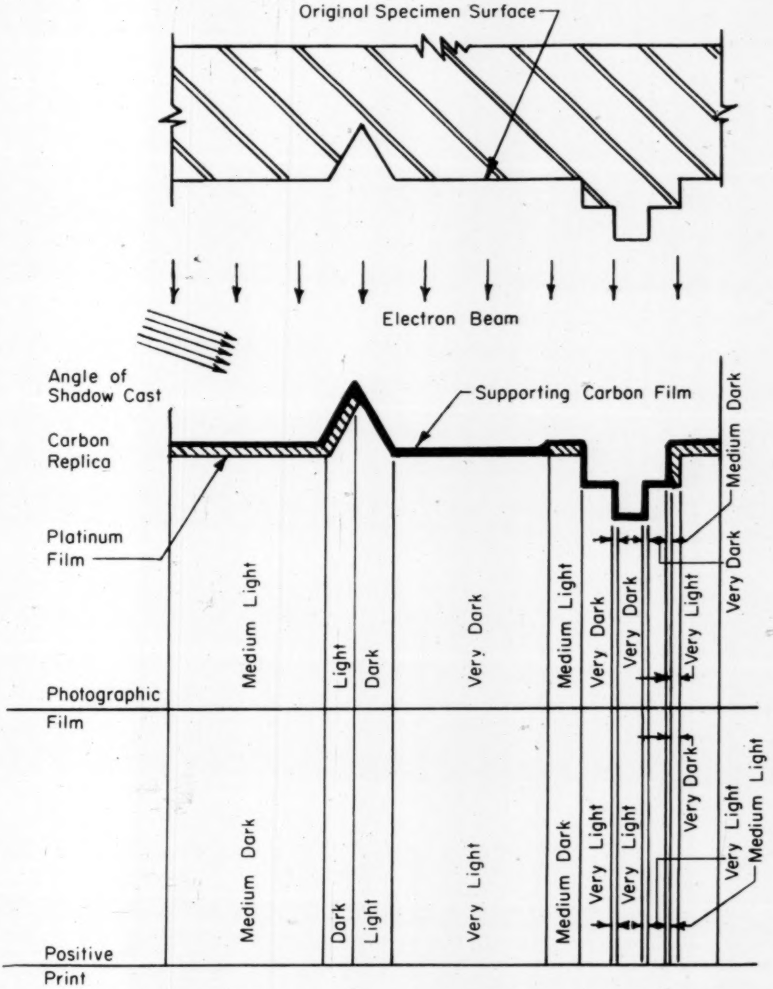
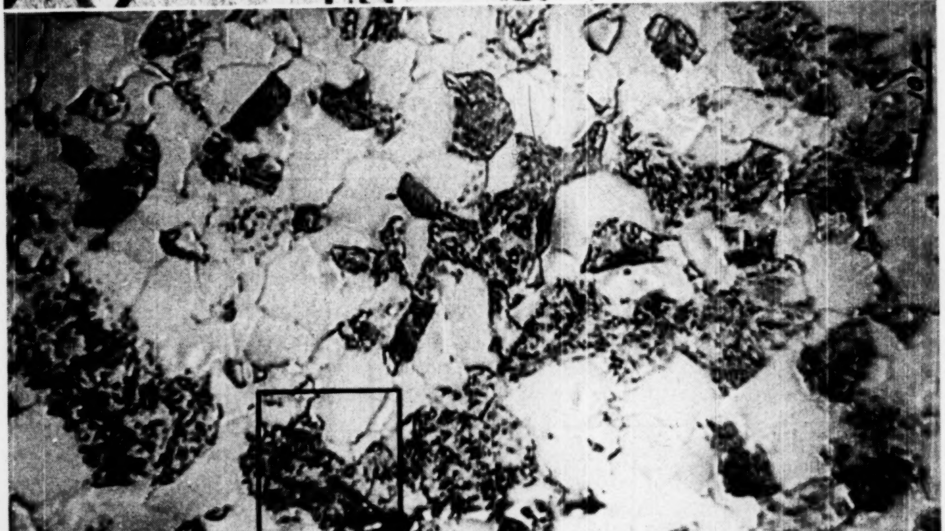
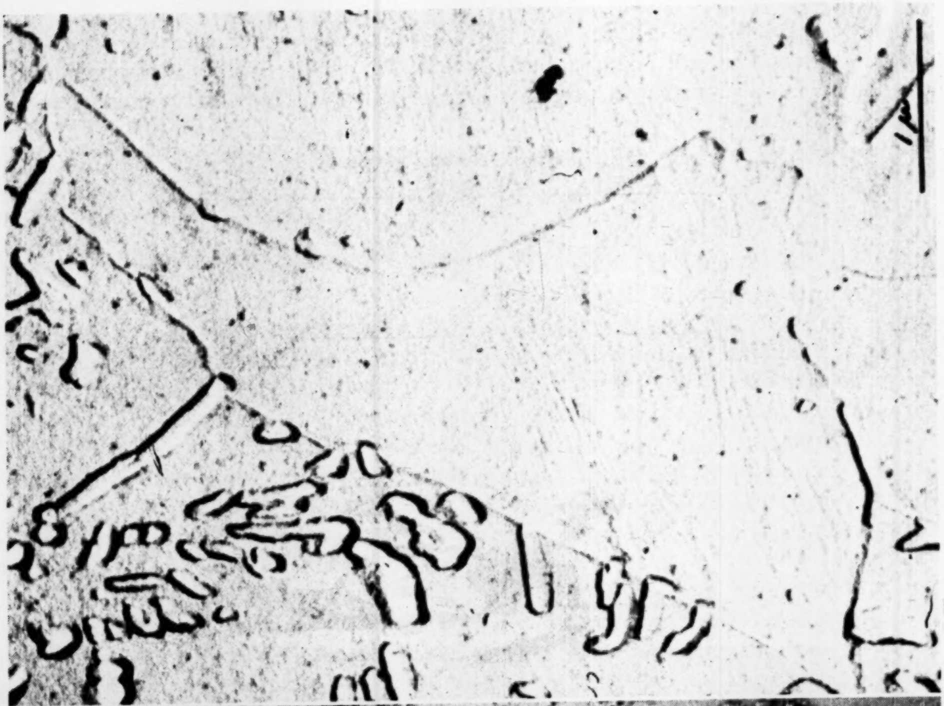

Original Specimen Surface
Electron Beam
Angle of Shadow Cast
Supporting Carbon Film
Carbon Replica
Platinum Film
Photographic Film
Positive Print
Medium Light
Light
Dark
Very Dark
Medium Light
Very Dark
Very Dark
Very Light
Very Dark
Medium Dark
Medium Dark
Dark
Light
Very Light
Medium Dark
Very Light
Very Light
Very Dark
Very Light
Medium Light

FIG. 1.—Schematic Diagram of Shadow-Cast Carbon Replica in Relation to Photographic Reproduction.

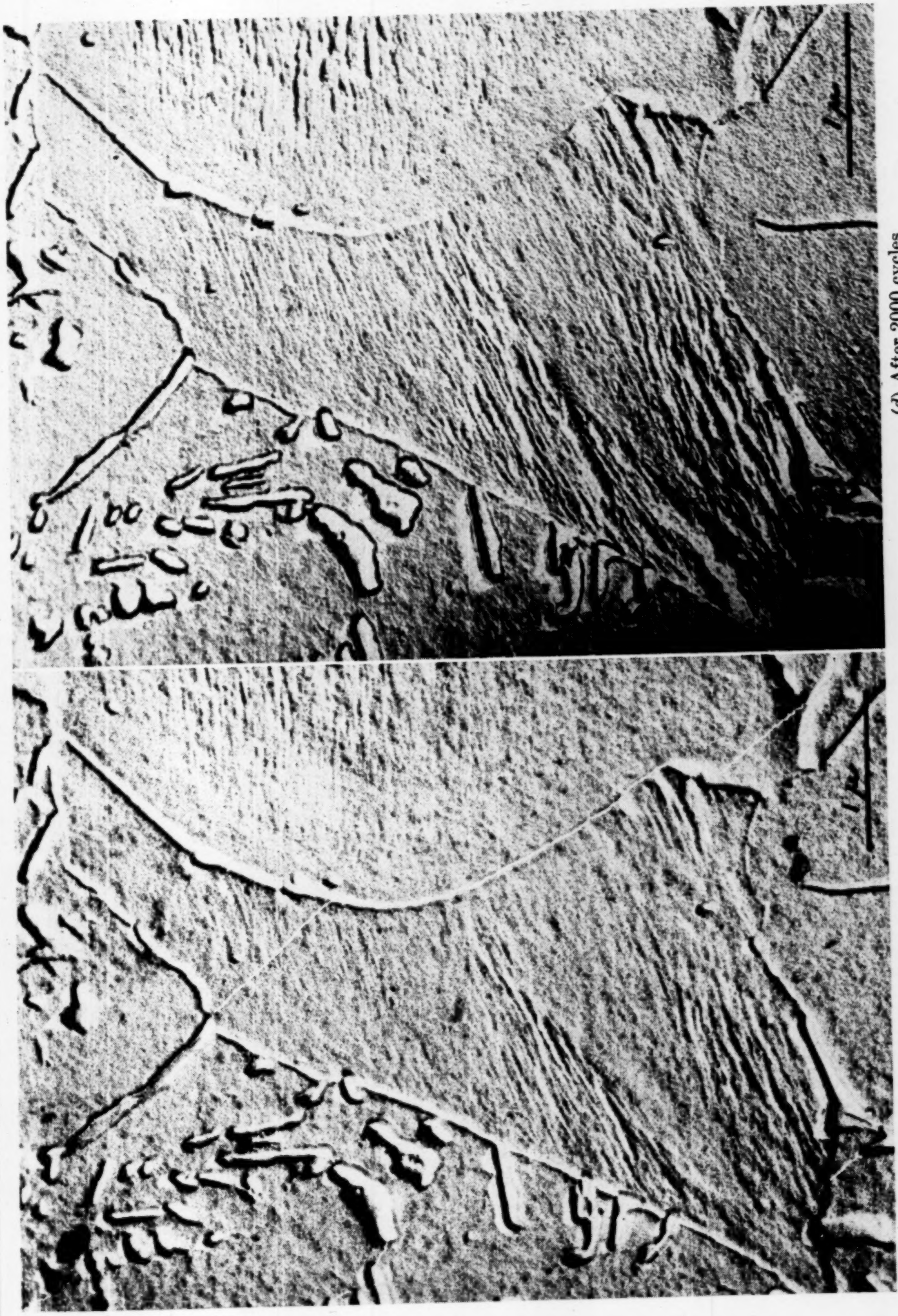
Axial fatigue tests were conducted using an SF-1-U Sontag fatigue machine. This machine was equipped with a magnifying level having a 5:1 ratio, so that a completely reversed load of up to



(*a*) Original surface



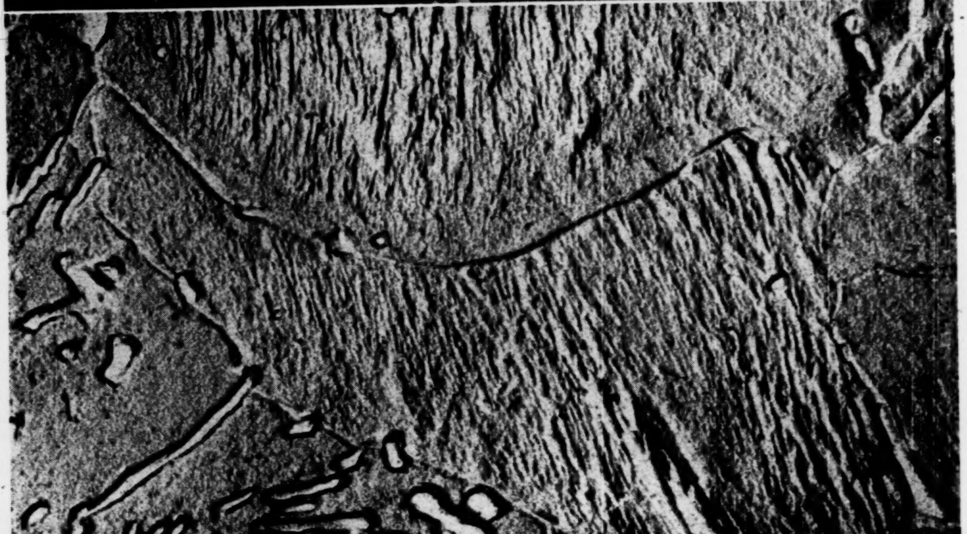
(*b*) After 500 cycles



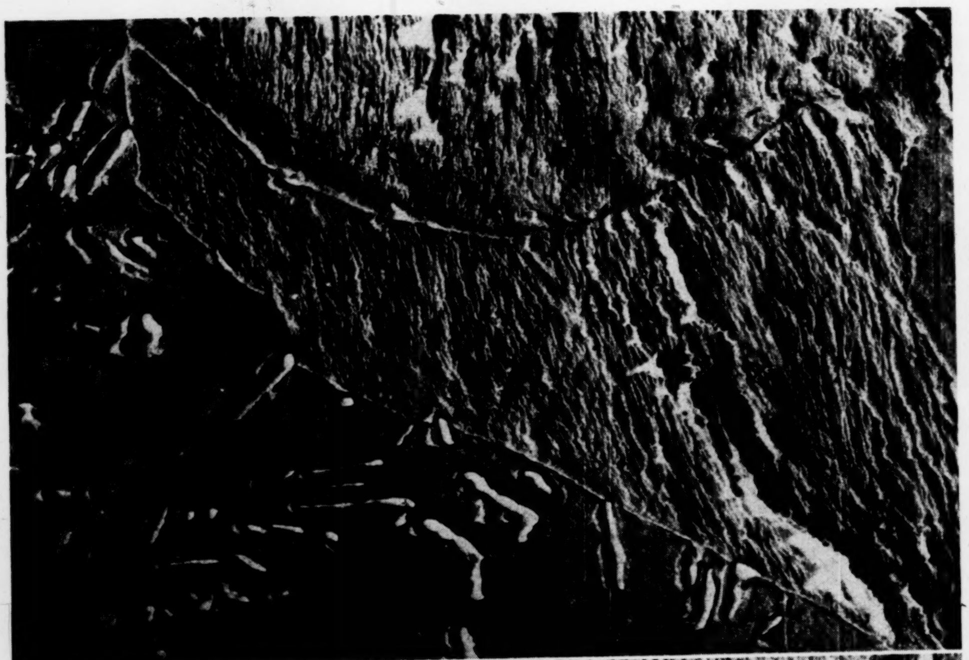
(c) After 1000 cycles

(d) After 2000 cycles

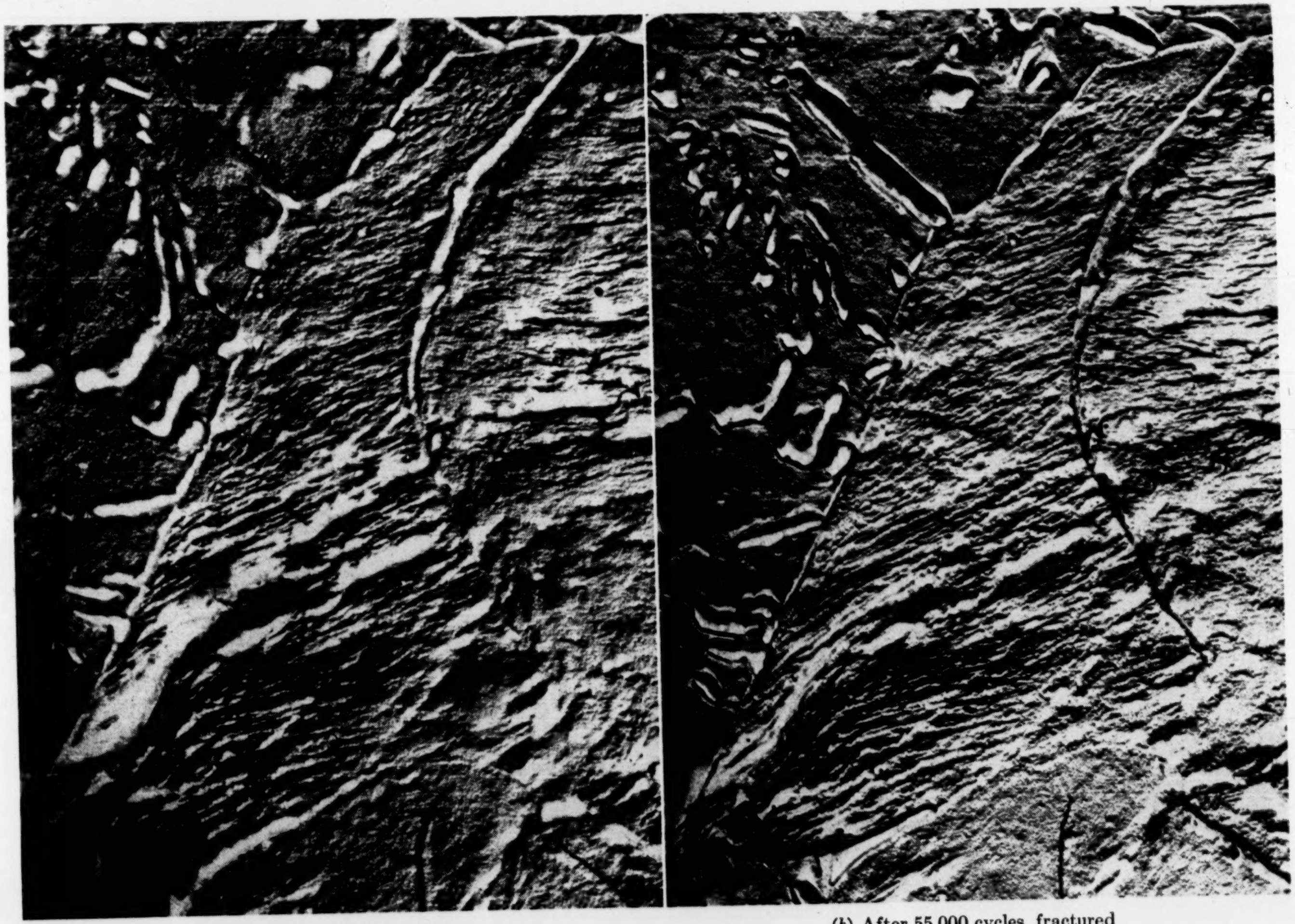
FIG. 2.—Surface Deformation Due to Alternating Stress of ±63,000 psi.



(e) After 5000 cycles



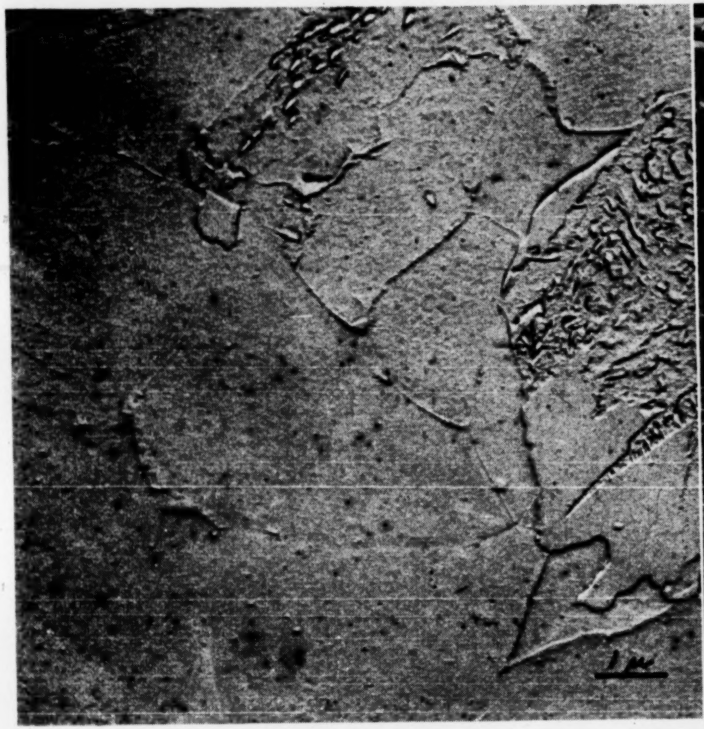
(f) After 20,000 cycles



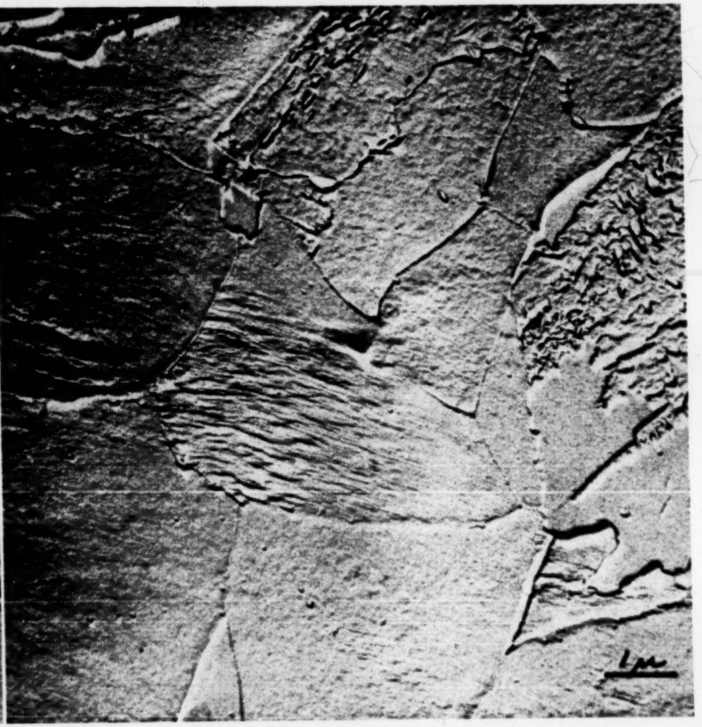
(g) After 40,000 cycles

(h) After 55,000 cycles, fractured

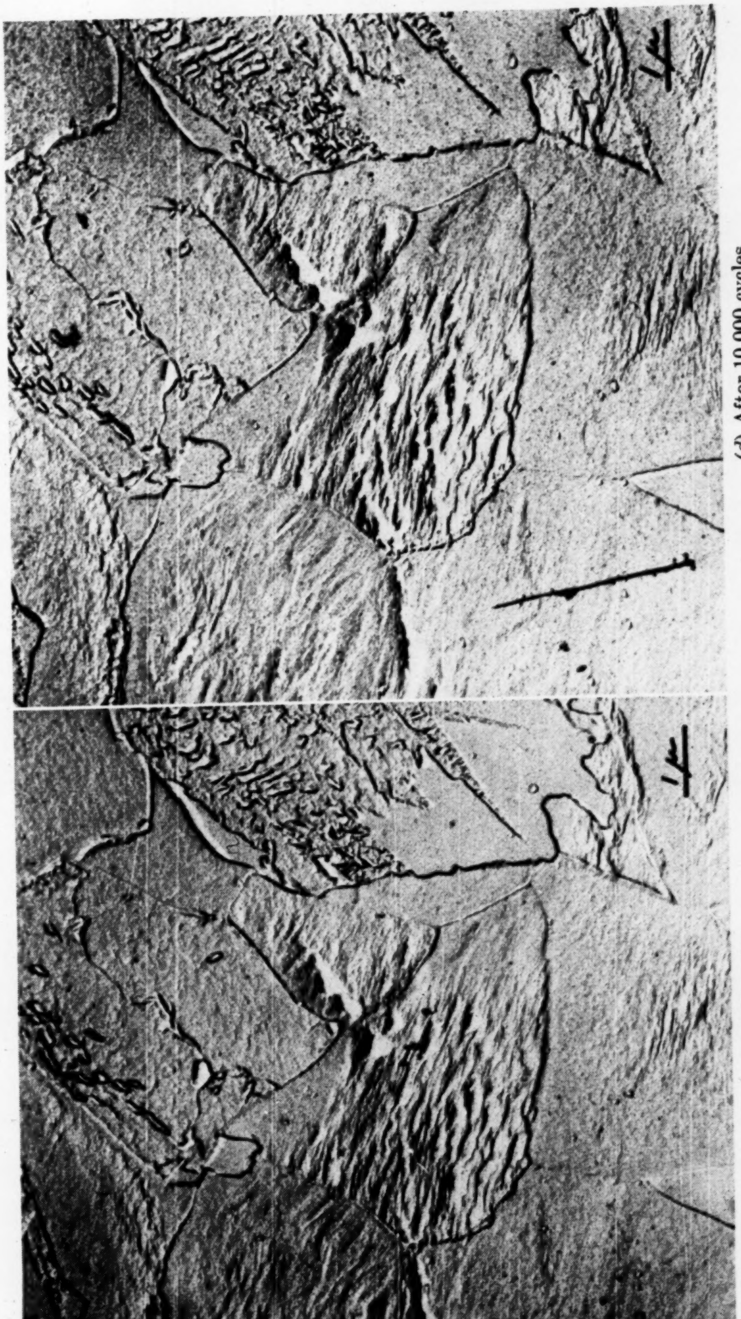
FIG. 2.—*Continued.*



(a) After 500 cycles



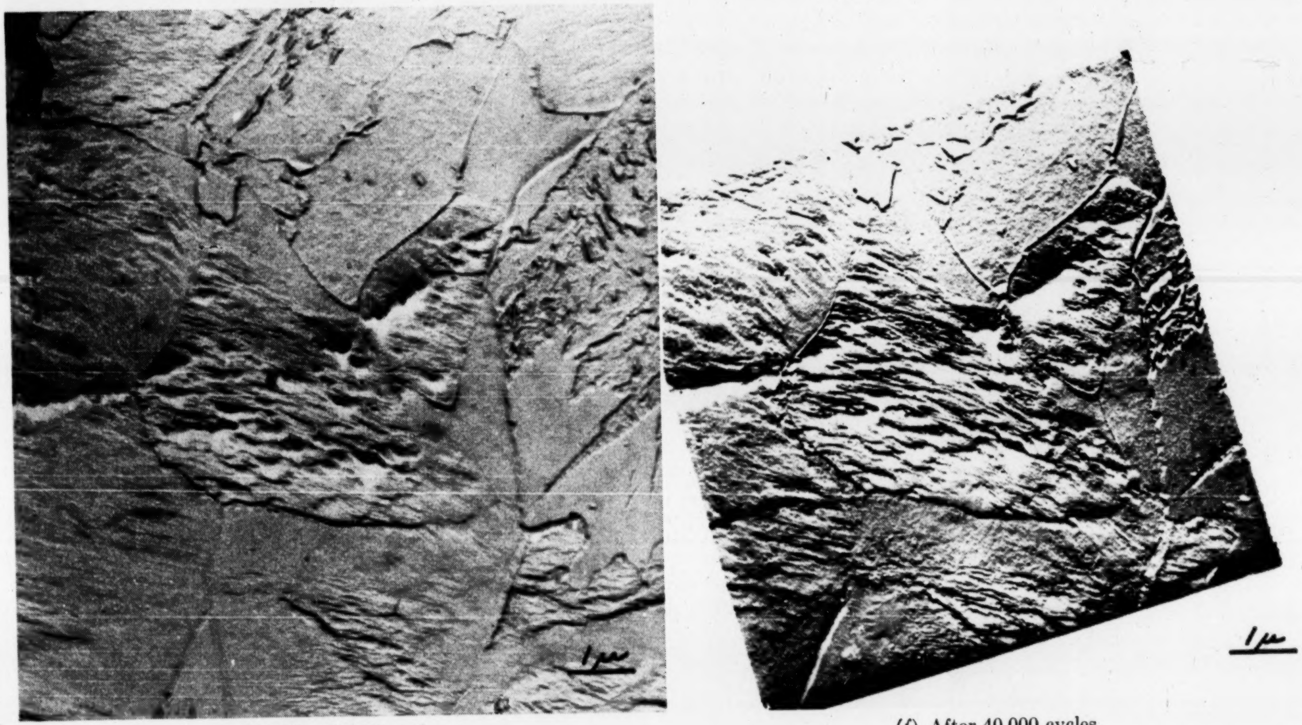
(b) After 2000 cycles



(c) After 5000 cycles

(d) After 10,000 cycles

FIG. 3.—Surface Deformation Due to Alternating Stress of ±63,000 psi.



(*e*) After 20,000 cycles

(*f*) After 40,000 cycles

FIG. 3.—*Continued.*

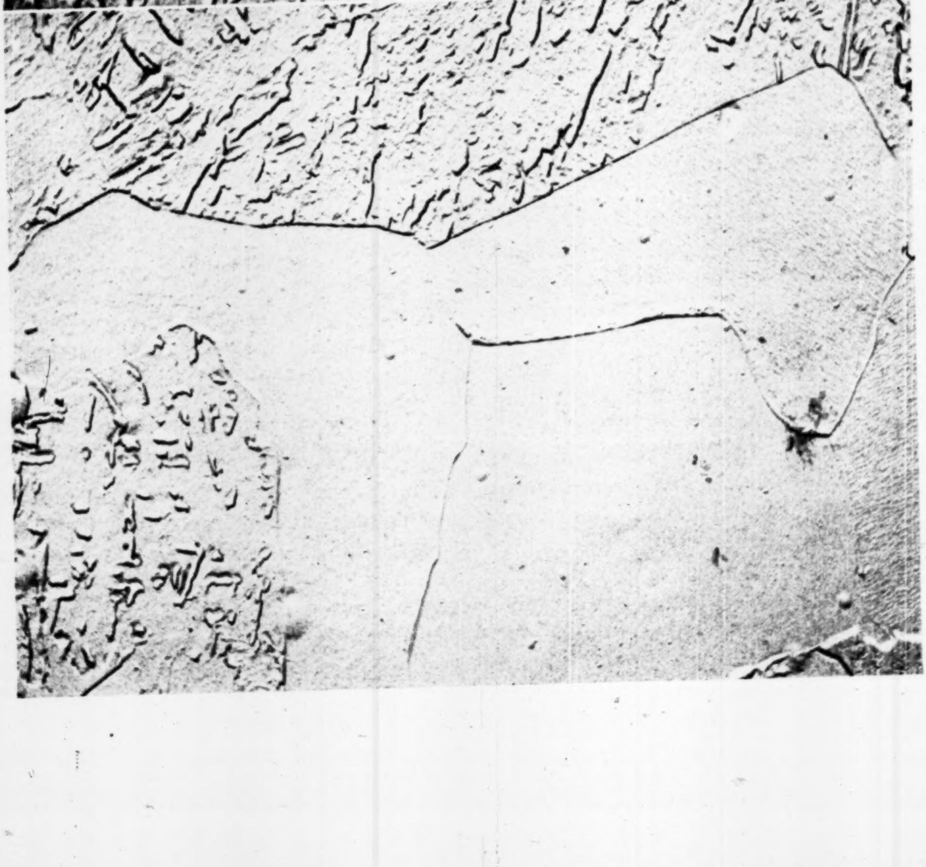
±5000 lb could be applied to the specimen. All the fatigue tests were conducted at a stress level of ±63,000 psi, as compared to an endurance limit of 53,000 psi for the material and specimen shape that was used. Low-temperature fatigue tests were performed by surrounding the specimen by a cup filled with liquid nitrogen. The liquid nitrogen that evaporated during the conduct of the test was continually replaced so as to maintain a constant temperature throughout the test. At intervals throughout the test, the specimen was removed from the fatigue machine, and a replica was made of the surface. For comparison of the surface deformation that occurs under a monotonically increasing load with that occurring under fatigue, one specimen was subjected to pure axial tension. This was accomplished by constructing a loading frame that would allow microscopic examination during load application. The loading frame was capable of retaining the load at various stress levels to allow a replica to be made of the specimen surface. This same procedure was used to determine the effects of a monotonically increasing load on a specimen previously damaged in fatigue.

REPLICA PROCEDURE

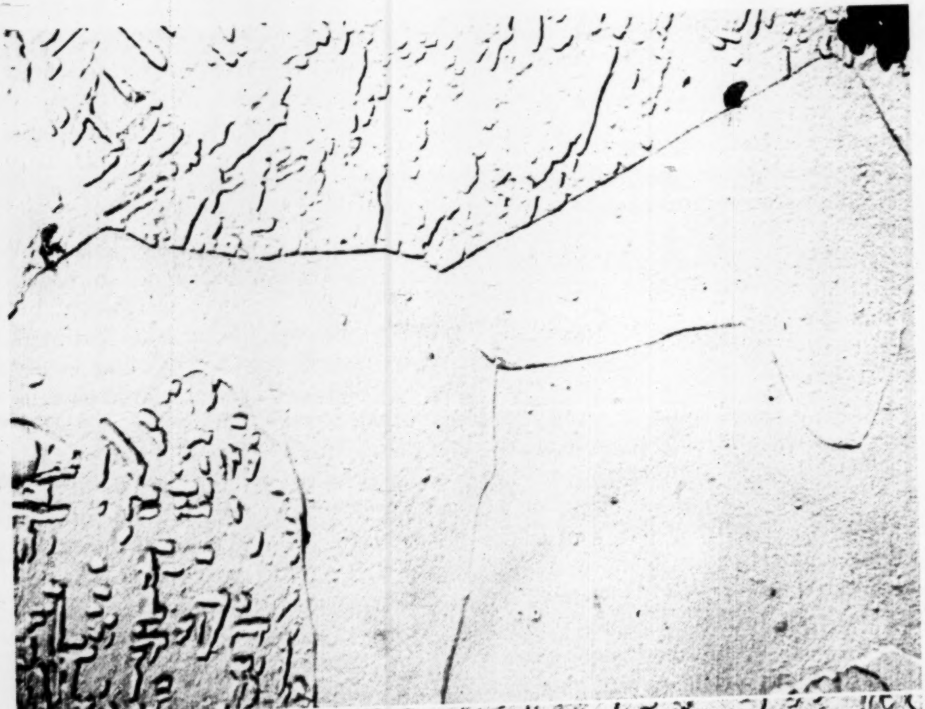
The electron microscope technique for the examination of metallographic structure will be described only briefly in this paper. The electron micrograph is made from a very thin replica of the specimen surface. The resolution of the image, or the ability to see fine detail, is mainly dependent upon the replica's ability to translate surface contours into a density contrast. The usual procedure for making a replica for metallurgical examination is to obtain a plastic cast of the surface of the specimen and to vacuum-deposit on the replica surface at a small incident angle a layer of a very dense metal. In this case, the dense metal was platinum. The platinum casts a shadow on the contours of the plastic matrix and provides the necessary density contrast. If the plastic matrix and platinum combination were put in the electron microscope, the electron beam would be scattered by the platinum and, to a lesser degree, by the plastic matrix. This scattering by the plastic base tends to reduce the resolution of the microscope by blurring the image. Because the platinum shadow is very thin (25 Å) and is too fragile to be used alone, a large gain in resolution can be obtained by replacing the plastic matrix by a thin carbon film (50 Å). Essentially, the carbon film is structureless and serves as a support for the platinum shadow. Figure 1 shows the completed replica in relation to the original specimen surface and the resulting positive photographic print. The density contrast on the left side of the replica would be representative of an intrusion in the specimen surface and that on the right side an extrusion. In the positive print, a crack would appear as a black line followed by a white shadow indicating its depth and shape. A more detailed description of this replica procedure as well as the technique that was used for selected-area examination will appear in a separate publication. There is evidence (11) to show that a carbon replica is capable of resolving less than 20 Å, providing a very sensitive method for fatigue damage detection.

TEST RESULTS AND THEIR INTERPRETATION

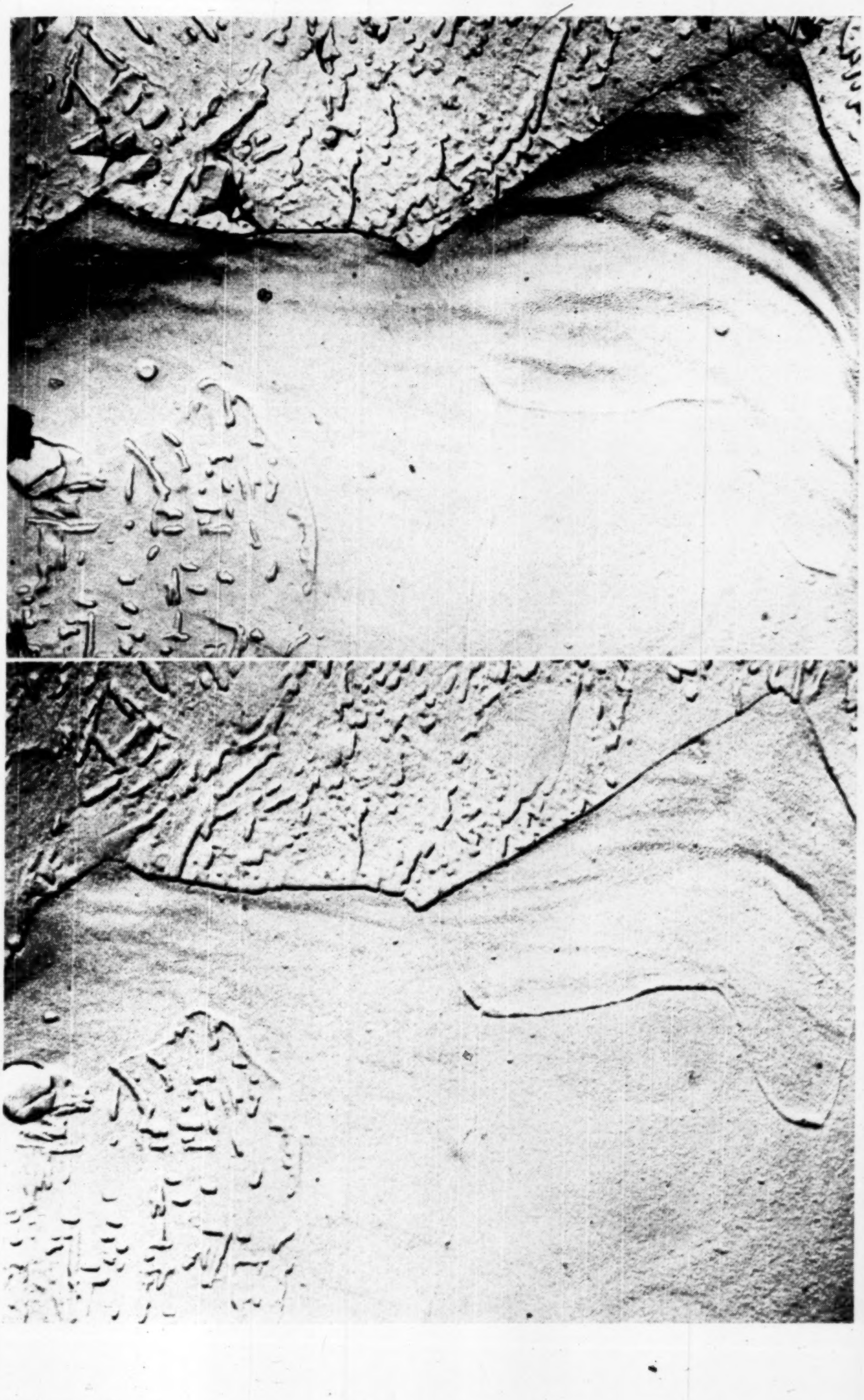
The damage occurring in a metallic crystal under the influence of a fatigue stress is shown in Figs. 2 and 3. Figure 2(*a*) shows an optical micrograph of the original specimen surface. The crystal selected for examination is shown in the center of the outlined area. The first appearance of damage is in the form of plastic slip in isolated regions of the



(*a*) Original surface



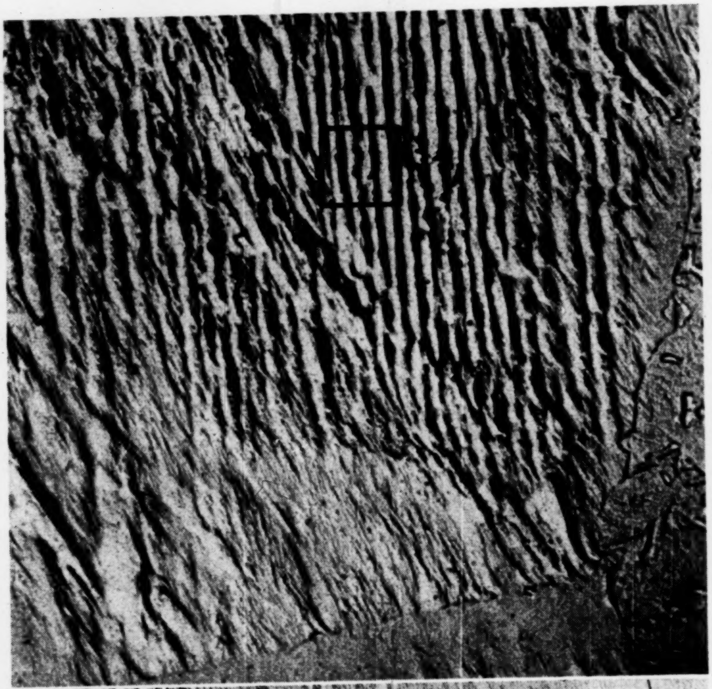
(*b*) After 1 per cent deformation



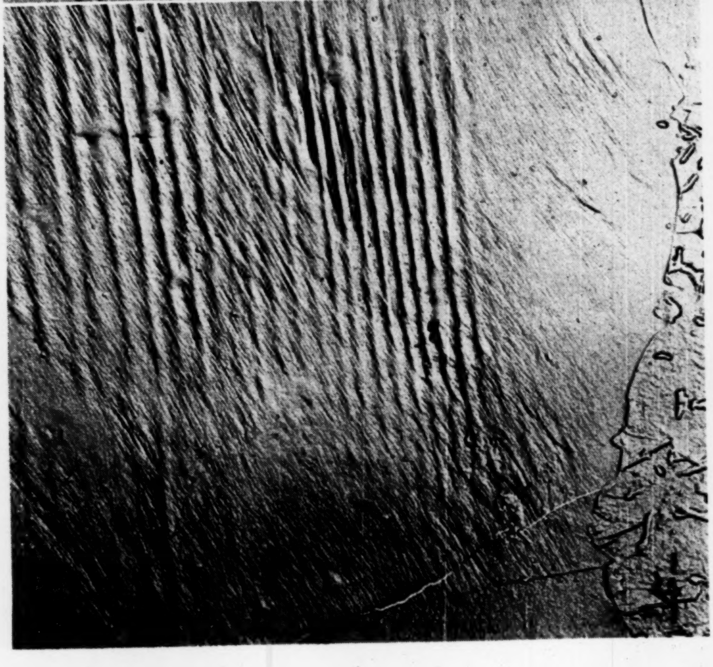
(c) After 6 per cent deformation

(d) After 10 per cent deformation

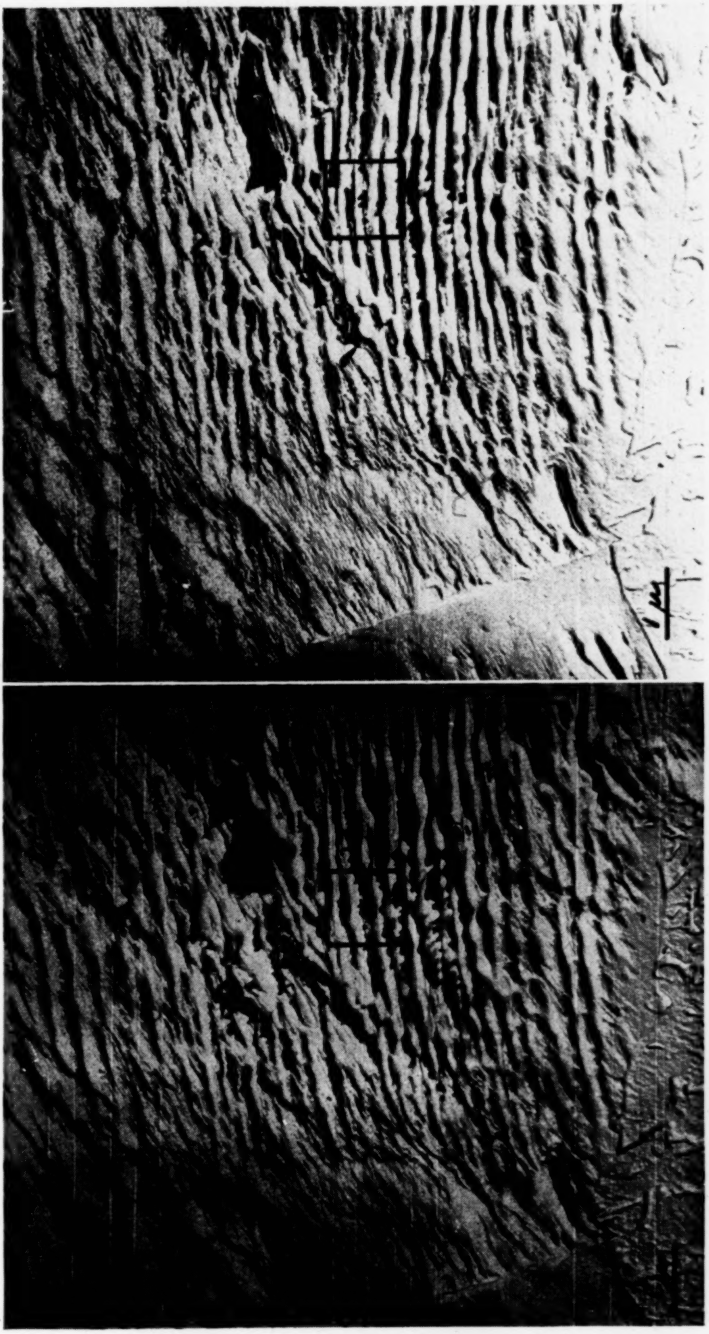
FIG. 4.—Surface Deformation Due to Static Tension.



(*b*) After 10,000 cycles at ±63 × 10^3 psi



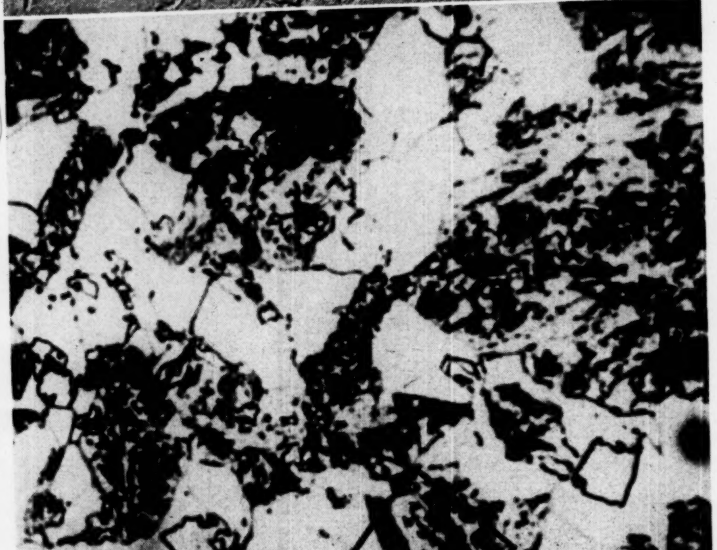
(*a*) After 2000 cycles at ±63 × 10^3 psi



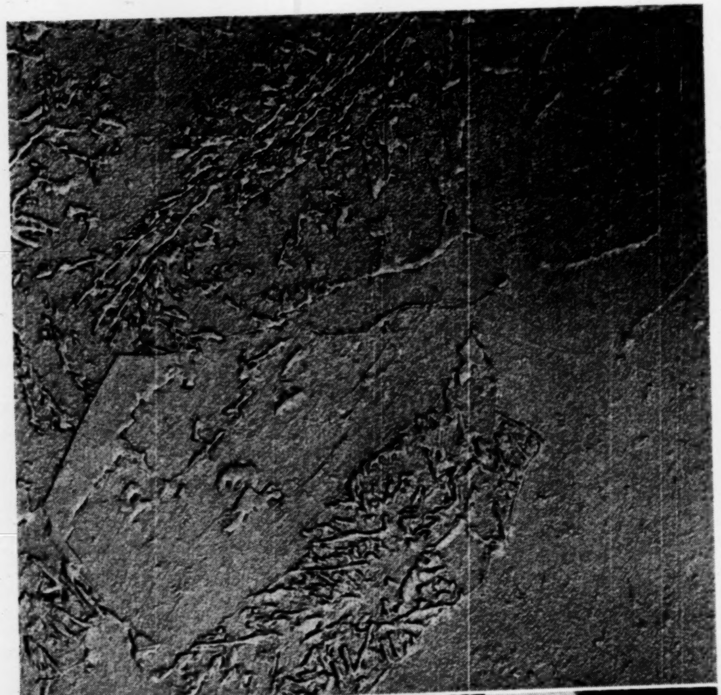
(*c*) After 10,000 cycles and static tested to 2.5 per cent deformation

(*d*) After 10,000 cycles and static tested to 5 per cent deformation

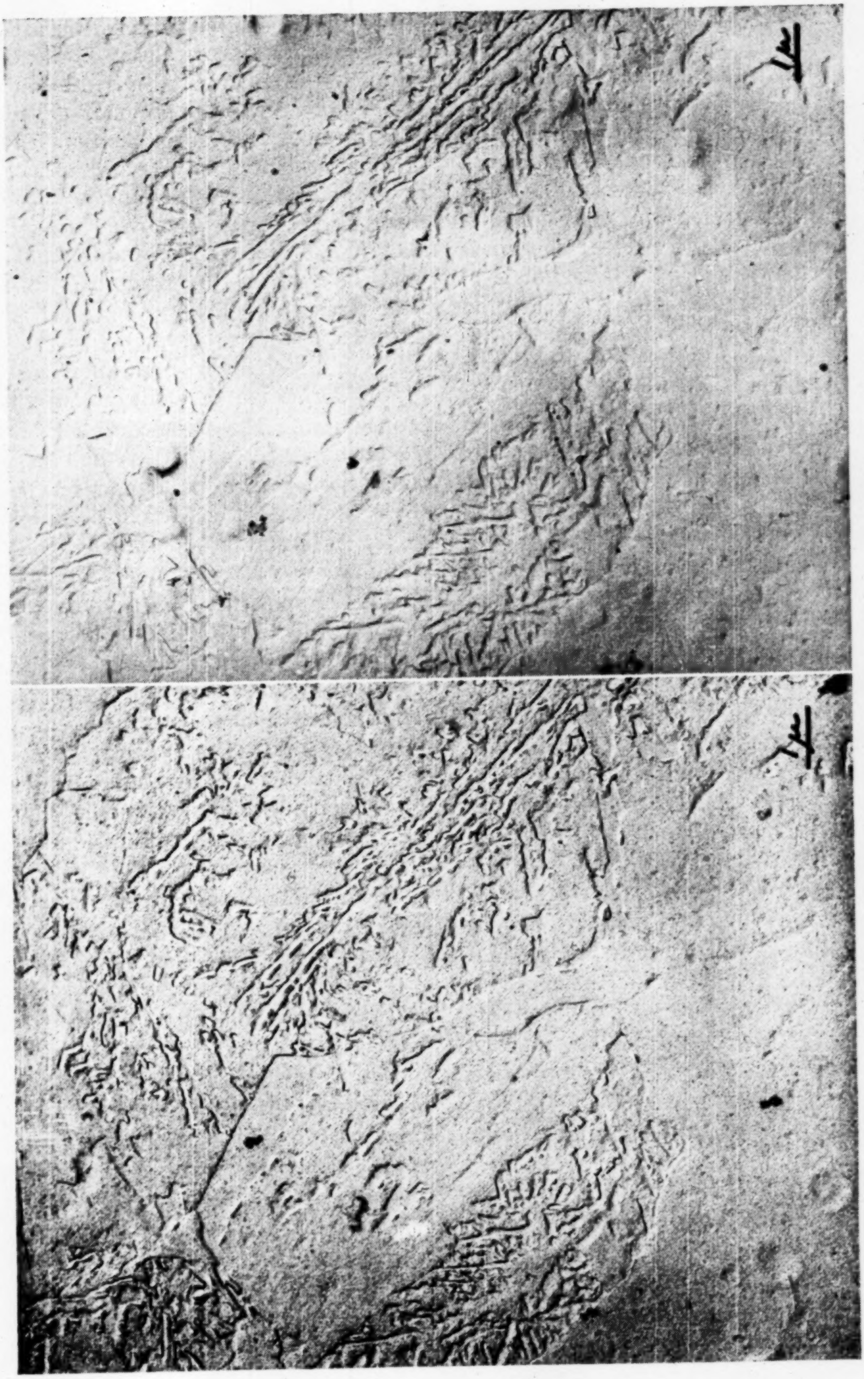
FIG. 5.—Surface Deformation Under Static Stress After Fatigue Precycling.



(*a*) Original surface (optical microscope)



(*b*) Original surface (electron microscope)



(c) After 5000 cycles at ±63 × 10^3 psi

(d) After 200,000 cycles at ±63 × 10^3 psi

FIG. 6.—Surface Deformation Due to Alternating Stress at −196 C.

ferrite crystal, Figs. 2(*a*) to (*d*). The slip lines that are formed appear to lie a fixed distance apart. Extensive examination of the specimen showed that slip lines did not form in the crystals containing the carbide precipitates. Increasing the number of cycles increases the number, length, and depth of the slip lines. The formation of cavities at the guide planes is shown in Fig. 2(*e*). Figures 2(*f*) and 2(*g*) show the cavity propagation leading to the formation of a microcrack. The black dots show the cavity positions and the white shadows show their shape. It is very possible that the formation of the cavity starts with the very first cycle, but owing to the limited resolution of the electron microscope, they remain undetected in the preceding electron micrographs. The cavities always seem to nucleate on the glide plane at a regular distance apart. This would possibly suggest some dislocation configuration acting as nuclei. The cavities also form preferentially in the areas of greatest plastic deformation. Figure 2(*f*) shows the result of cavity propagation, a set of discrete points which could be defined as an incipient microcrack. The actual microcrack is shown in Fig. 2(*g*). In Fig. 2(*h*), which was made after fracture at 55,000 cycles, the once very prominent cracks disappear. The only explanation that can be given is that the material contained residual stresses, which were relieved at fracture. A similar sequence of pictures, Figs. 3(*a*) to (*f*), representing another test specimen, shows the cavity formation leading to the formation of a microcrack.

Based upon this evidence, it would be logical to assume that the mechanism of cavity formation at room temperature is one of diffusion—specifically, stress-motivated diffusion of vacancies produced internally in the crystal through the action of dislocations. Dislocation models given by Read (**12**) and Dorn (**13**) demonstrate the feasibility of certain dislocation configurations acting as a source of vacancies. Experimental verification of this idea by Keith and Gilman (**6**) shows that reversed dislocation motion leaves behind a trail of point defects—vacancies or interstitial atoms. Diffusion mechanisms of this type have been noted by Balluffi and Seigle (**14**) for grain-boundary voids in creep and by Brinkman (**15**) for void formation during Kirkendall-type diffusion.

There still remain some questions as to the formation of cavities and their propagation. The first question that should be asked is whether the creation of cavities is present only under reversed-stress conditions. The second is whether cavity propagation is caused by the annihilation of vacancies or whether it is due to the breaking of the atomic bonds at the corners of the cavity because of the high stress concentration. The third question is concerned with the extreme temperature dependence of any diffusion phenomena. If the proposed mechanism is correct, then at a temperature approaching absolute zero fatigue should not be present. McCammon and Rosenberg (**16**) have shown that this is not the case with face-centered cubic crystals. However, there still remains the possibility of another mechanism prevailing at these low temperatures. Figure 4 illustrates the crystallographic deformation that occurs under the action of a monotonically increasing stress. Here the slip lines formed are much finer in texture, compared to those formed during stress reversal. The individual slip motions are beyond the resolution ability of the microscope, and as such the slip aggregations appear as coarse lines on the electron micrographs. At no time during the test were any microcracks observed in the material, nor were any cavities formed. The maximum local plastic deformation that was examined was 10 per cent, because the roughness of the surface made it almost impossible to obtain good plastic replicas.

In order to answer the second question concerning the mechanism of cavity propagation, a specimen was damaged in fatigue and then subjected to static tension. (Fig. 5). A degree of damage was chosen such that isolated cavities existed on the surface after 10,000 cycles at a stress level of ±63,000 psi. This damage is illustrated in Fig. 5(*b*). Upon applying a static tensile stress, it was found that the cavities became deeper and were spread open by the tensile stress; however, these cavities did not grow appreciably larger. Furthermore, a series of discrete points remained discrete and did not propagate to form a continuous crack. Even after microscopic plastic deformations of 15 per cent, no new slip fields or lines became visible nor did any new cavities form (area enclosed by square). Because the small microcrack surface cavities do not propagate under a static tensile load, their propagation under a cyclic load must be caused by their acting as a sink for vacancies rather than by rupture at the edges due to stress concentration. This fact was also confirmed experimentally by Marin *et al* (**17**) from the fact that there were no changes in the macroscopic mechanical properties due to a fatigue precycle.

In order to determine the thermal dependence of the cavity formation mechanism, a fatigue test was performed at the same stress level, ±63,000 psi, but at a temperature of −196 C. As was predicted, the symptoms of fatigue damage did not appear. As shown in Fig. 6, the surface of a typical crystal was unchanged after 200,000 cycles. There were no slip lines formed; thus the strain displacement itself was insufficient to cause dislocation motion. If fatigue fracture were to take place at this temperature, it would have to be caused by some other type of crystal imperfection.

CONCLUSIONS

The following conclusions can now be drawn concerning the mechanism of crack formation during fatigue. During the period of stress application, cavities form on the surface of the crystal and propagate until adjacent cavities meet and form a microcrack. The formation mechanism is one of diffusion of vacancies from some internal source. This source may be the suitable motion of a jog formed by the intersection of a screw and an edge dislocation. The cavities enlarge by acting as sinks for the vacancy currents. There still remains the question of a suitable nucleus for the cavity. This may be some dislocation configuration or an initial imperfection on the crystal surface. A further study of this phase should be rewarding. There exists the possibility of the grain boundaries acting as a sink for the vacancies, although this did not appear to be evident at the surface. Further tests are contemplated in which sections will be taken to investigate any internal grain-boundary motion or void formation.

In order to correlate this mechanism with observed macroscopic data, one must define the endurance limit. This would be defined as a threshold value of stress below which the vacancies created would be absorbed by imperfections in the crystal volume, such as inoperative dislocations, interstitial atoms, etc. At any stress above this value, a steady-state concentration of vacancies would exist. This concentration of vacancies would be a function of the stress level above the endurance limit.

REFERENCES

(1) H. J. Gough, "Crystalline Structure in Relation to the Fatigue of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 33 (II), p. 1 (1933).

(2) F. P. Bullen, A. K. Head, and W. A. Wood, "Structural Changes During Fatigue of Metals," *Proceedings*, Royal Soc. (London), Vol. 216, Series A, p. 322 (1953).

(3) P. J. H. Forsyth, "Some Further Observations on the Fatigue Process in Pure Aluminum," *Journal*, Inst. Metals, Vol. 82, p. 449 (1954).

(4) M. Hempel, "Uber Verformungerscheinungen in Stahlen bei Wechselbeanspruchung," Colloquium on Fatigue, Int. Conference of Theoretical and Applied Mechanics, Stockholm (1956).

(5) W. A. Wood, and A. K. Head, "Some New Observations on the Mechanism of Fatigue in Metals," *Journal*, Inst. Metals, Vol. 79, April 1951.

(6) R. M. Keith and J. J. Gilman, "Progress Report on Dislocation Behavior in Lithium Fluoride," Symposium on Basic Mechanisms of Fatigue, Am. Soc. Testing Mats., p. 21 (1958). (Issued as separate publication *ASTM STP No. 237*.)

(7) N. Thompson, "Experiments on the Origin of Fatigue Cracks," in Fatigue in Aircraft Structures, Academic Press (1956).

(8) W. J. Craig, "An Electron Microscope Study of the Development of Fatigue Failures," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 877 (1952).

(9) S. J. Love, "Structural Changes in Ingot Iron Caused by Plastic and Repeated Stressing," *Technical Report No. 33*, on the Behavior of Materials Under Repeated Stress, Dept. of Theoretical and Applied Mechanics Univ. Ill., Urbana, Ill. (1952).

(10) W. A. Wood, "Recent Observations of Fatigue Failure," Symposium on the Basic Mechanisms of Fatigue, Am. Soc. Testing Mats., p. 110 (1958). (Issued as separate publication *ASTM STP No. 237*.)

(11) D. E. Bradley, "A High Resolution Evaporated-Carbon Replica Technique for the Electron Microscope," *Journal*, Inst. Metals, Vol. 83, p. 35 (1954).

(12) W. T. Read, "Dislocations in Crystals," McGraw Hill Book Co., Inc., New York, N. Y. (1953).

(13) J. E. Dorn, "Basic Aspects and Physical Theories of Creep of Metals," Proceedings for a Short Course in Materials Engineering Design for High Temperatures, Pennsylvania State Univ., University Park, Pa. (1958).

(14) R. W. Balluffi and L. L. Seigle, "Growth of Voids in Metals During Diffusion and Creep," *Acta Metallurgica*, Vol. 5, p. 449 (1957).

(15) J. A. Brinkman, "Mechanism of Pore Formation Associated with the Kirkendall Effect," *Acta Metallurgica*, Vol. 3, p. 140 (1955).

(16) R. D. McCammon and H. M. Rosenberg, "The Fatigue and Ultimate Tensile Strengths of Metals Between 4.2 and 293 Kelvin," *Proceedings*, Royal Soc. (London), Vol. 242, p. 203 (1957).

(17) J. Marin, P. Borachia, and U. A. Rimrott, "The Effect of Stress Cycling on the Static Mechanical Properties of SAE 4340 Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 662 (1959).

DISCUSSION

Mr. George J. Danek, Jr.[1]—From everything I have read on this recently I have the impression that these first observable cracks occur very early in the fatigue life; some say within 5 or 10 per cent. Would the author please comment on this?

Mr. Melvin A. Wilkov (*author*).—I would comment this way. It depends upon the stress condition. If a stress distribution in a specimen is so oriented that a maximum occurs on the surface as in bending or at a point on the surface of the specimen as at a notch or surface imperfection, we would be able to observe both the formation of the crack as well as the propagation of the crack. The presence of microcrack after 5 or 10 per cent of the specimen life would then most likely be after the crack has formed and some crack propagation mechanism has started.

In the studies I have undertaken, axial tension-compression, we have a more uniform stress field. Once a microcrack has been formed it provides the added stress concentration to provide a very rapid propagation. Fracture takes place almost immediately after the microcrack has formed.

Mr. L. F. Coffin, Jr.[2]—Do the void formations observed in slip bands occur predominantly at the surface of the specimens, or are they distributed throughout the bulk of the stressed metal? To what depth do individual voids penetrate into the surface?

Mr. Wilkov.—It would be possible that the void formation takes place on any surface including the internal grain boundaries. Up to the present time I know of no nondestructive method to observe any internal void formations in the body. However, if the fatigue-damaged specimen surface were repolished to a depth of 1μ, removing all cavities formed, the specimen would revert to its virgin condition as far as the formation of new cavities was concerned. This has been shown by others including Thompson,[3] so it leaves one to speculate that the void formation is an external surface phenomenon. There is, however, a higher stress magnitude on the external surface which may account for this fact.

---

[1] High-Temperature Alloys Branch, Metallurgy Division, Naval Research Laboratory, Washington, D. C.

[2] Research Laboratory, General Electric Co., Schenectady, N. Y.

[3] N. Thompson, N. Wadsworth, and N. Louat, *Philosophical Magazine*, Vol. 1, p. 113 (1956).

THE R. R. MOORE ROTATING-BEAM FATIGUE TEST*

THE EFFECT OF CHANCE VARIATIONS IN SPECIMEN PREPARATION ON THE FATIGUE STRENGTH OF COLD-DRAWN ALLOY A PHOSPHOR BRONZE ROD

BY G. R. GOHN[1] AND A. FOX[1]

SYNOPSIS

Rotating-beam fatigue tests using R. R. Moore type specimens prepared in accordance with the information given in the ASTM Manual on Fatigue Testing (**1**)[2] have been used for many years to evaluate the fatigue properties of various materials. However, the authors' tests indicate that the information given on specimen preparation is inadequate for fatigue tests on strain-hardenable materials such as phosphor bronze rod. Consequently, the rotating-beam fatigue test becomes a measure of the effect of residual stresses resulting from the methods used in preparing the test specimens—particularly the polishing operation—rather than a determination of the fatigue properties of the material. Obviously, such findings indicate that some revision in the text of the ASTM Manual on Fatigue Testing is required to correct this situation.

Fatigue tests to obtain *S-N* diagrams for materials are made with test specimens of relatively simple design for each product. Thus for tests on rod, bar stock, and plate the simple rotating-beam specimen, commonly known as the R. R. Moore type, has been accepted and used for years by many laboratories; in fact it is probably more widely used than any other type of fatigue test specimen. Recommended dimensions for the R. R. Moore specimen are given in Fig. 9 of the ASTM Manual on Fatigue Testing (**1**),[2] and certain of the instructions for its preparation are quite specific, others are less detailed.

The instructions covering the preparation of the simple unnotched rotating-beam fatigue specimen (the R. R. Moore type) state that "procedures for preparing the portion of the test specimen on which the calculated fatigue stresses are imposed require standardization to permit correlation of results between laboratories and to obtain comparable and reproducible values." The instructions specify details for the machining operation which "will minimize eccentricity in the finished product" and require that the specimen shall be finished "with a sharp tool and light cuts to prevent bending, overheating, or cold working." A speed of 500 rpm with a feed of 0.0015 in. is suggested. In the case of material having a hardness greater than approximately Rockwell C 40, grinding rather than machining is specified. In either case, 0.003 to 0.005 in. on the diameter is allowed for final polishing. No details are, however, given for either the grinding or the polishing operation.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The foregoing instructions imply that, if fatigue test specimens are made from the same lot of material either by different machine shops or at different times by the same shop, the test results should be comparable. Our experience fails to justify this conclusion. Instead, we find that the instructions on specimen preparation appear to be inadequate, and the use of such test specimens may lead to mistaken conclusions regarding the fatigue characteristics of the material under test.

REVIEW OF THE LITERATURE

Fatigue investigators are well aware that, in bending, the fatigue strength is primarily a function of the surface or subsurface conditions. The beneficial effects of such operations as shot peening, which leave the surface with residual compressive stresses, and the injurious effects of the normal electroplated finishes, which leave the surface with high residual tensile stresses, are too well known to require documentation. Recent articles have discussed the effect of surface roughness (**2–11**), polishing (**4,5,7,8, 12–14**), and grinding (**4,5,8,9,12,15–18**) on fatigue life. While these authors discuss the effect of various surface finishes on fatigue life, none of them deal with the problem in hand, namely, whether the variations in the surface finish inherent in the preparation of fatigue test specimens as prescribed in the manual affect the test results. Or, in other words, does use of such specimens insure comparable and reproducible test results and permit correlation of results between laboratories? A review of the authors' fatigue data will provide the answer to these questions.

ORIGIN OF INVESTIGATION

To obtain basic fatigue data for use in evaluating some more sophisticated high-speed, short-time fatigue tests, the authors undertook some rotating-beam fatigue tests on R. R. Moore testing machines. The test specimens were unnotched cylindrical specimens obtained from two commercial machine shops currently engaged in preparing fatigue test specimens for use in various university and governmental agency research projects. Both shops were requested to finish the specimens, from phosphor bronze rod supplied by the authors, by machining and polishing in accordance with the information given in Fig. 9 and section IV of the manual. The diameter specified by the authors for the finished specimens was 0.300 ± 0.001 in., the radius approximately 9 in.

MATERIALS TESTED

The materials tested were two lots of $\frac{1}{2}$-in. diameter, hard-drawn alloy A (5 per cent tin) phosphor bronze rod made by the Riverside Alloy-Metal Div., H. K. Porter Co., Inc., Riverside, N. J. The mechanical properties of these two rods are described in a paper by Gohn and Bader (**19**). One lot, designated No. 1, was cold-drawn 27 per cent after a 0.035-mm ready-to-finish grain size anneal; the other, designated No. 2, after a 0.008-mm ready-to-finish anneal.

TEST PROGRAM AND DATA

To evaluate the fatigue properties of the hard-drawn phosphor bronze rod, a series of *S-N* diagrams was developed from tests on specimens obtained from two machine shops. In addition, a nonferrous research laboratory, which normally conducts its fatigue studies on unpolished specimens, prepared a set of R. R. Moore specimens from the 0.008-mm rod. A second set of unpolished specimens was obtained from a commercial testing laboratory that normally does not make R. R. Moore specimens. This was done to see what variation in fatigue results might be expected in tests on unpolished specimens obtained from two different sources. Later, one of the commercial machine shops prepared a set of unpolished specimens from the 0.008-mm material and a duplicate set of machined and polished specimens. Half of each of the last two groups of specimens were stress-relief annealed for 4 hr in hydrogen at 400 F prior to test. Each lot was checked to determine the quality of the surface finish. For this purpose a Brush surface analyzer was used. Four Baldwin-Lima-Hamilton R. R. Moore type fatigue machines were used to obtain the test data.

All four machines were checked for bearing concentricity and taper, the latter being corrected so that it was identical on all eight bearings. Specimen runout in the assembled bearings was also checked prior to test, the total indicator movement being no greater than 0.002 in. A summary of the test program and the fatigue data are shown in Table I.

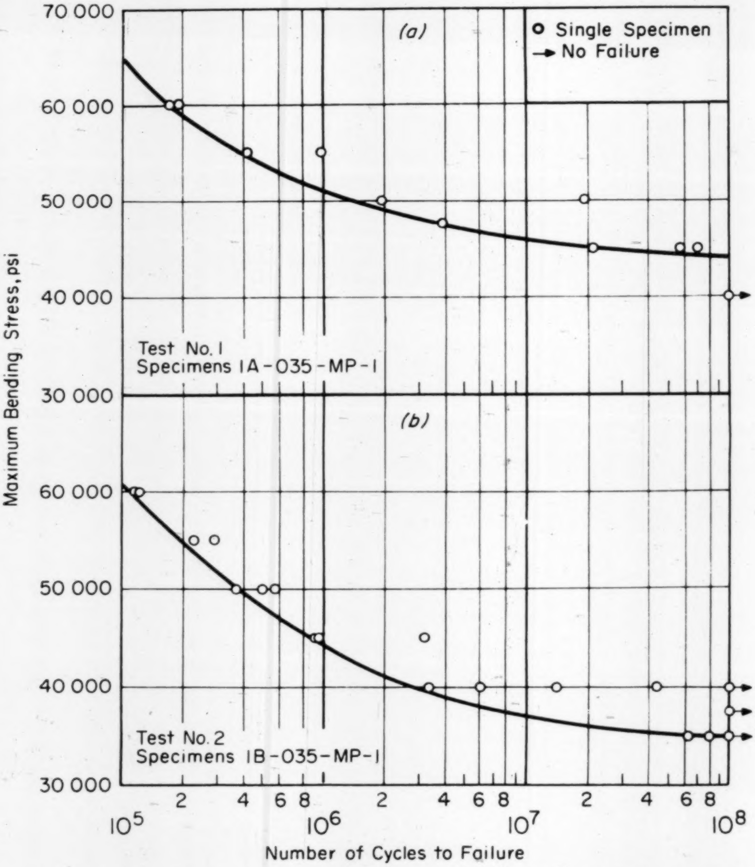


FIG. 1.—Fatigue Characteristics of Hard-Drawn Alloy A Phosphor Bronze Rod.
R. R. Moore specimens. Diameter = 0.300 in. Nominal ready-to-finish grain size = 0.035 mm.

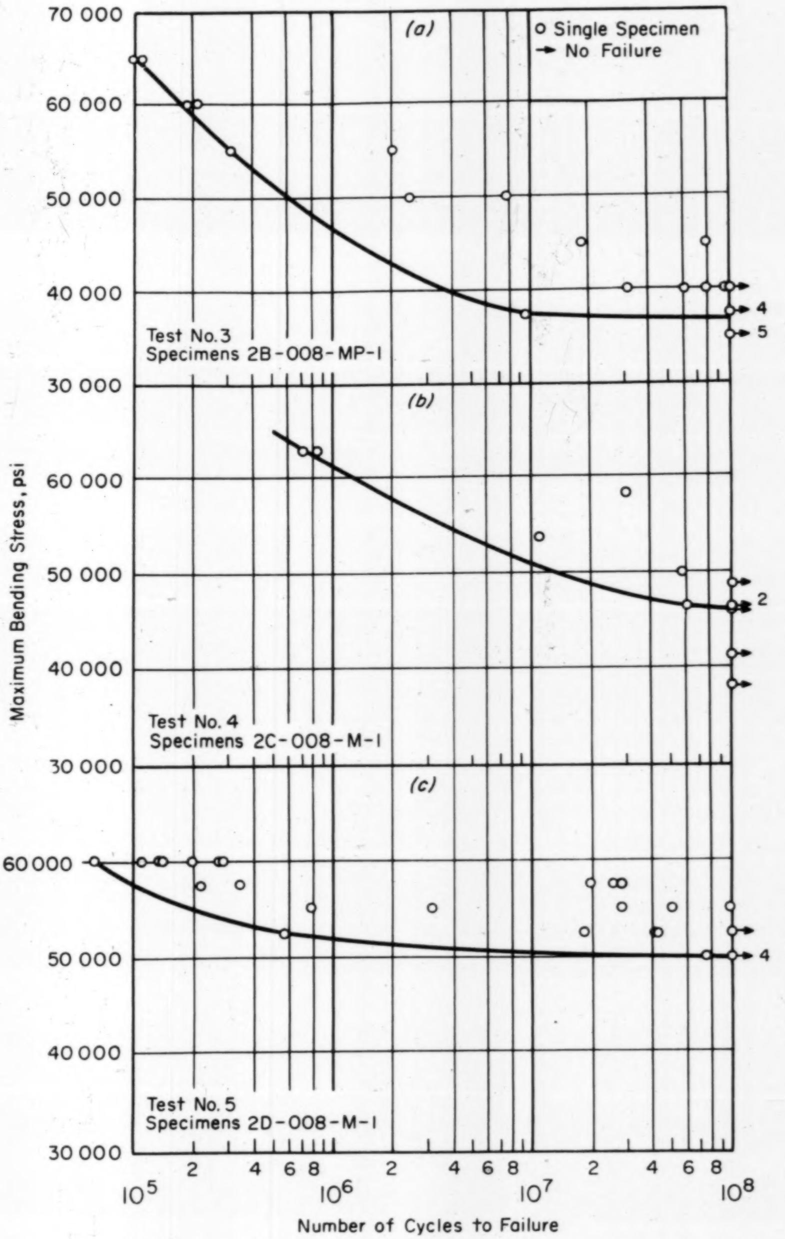


FIG. 2.—Fatigue Characteristics of Hard-Drawn Alloy A Phosphor Bronze Rod.
R. R. Moore specimens. Diameter = 0.300 in. Nominal ready-to-finish grain size = 0.008 mm

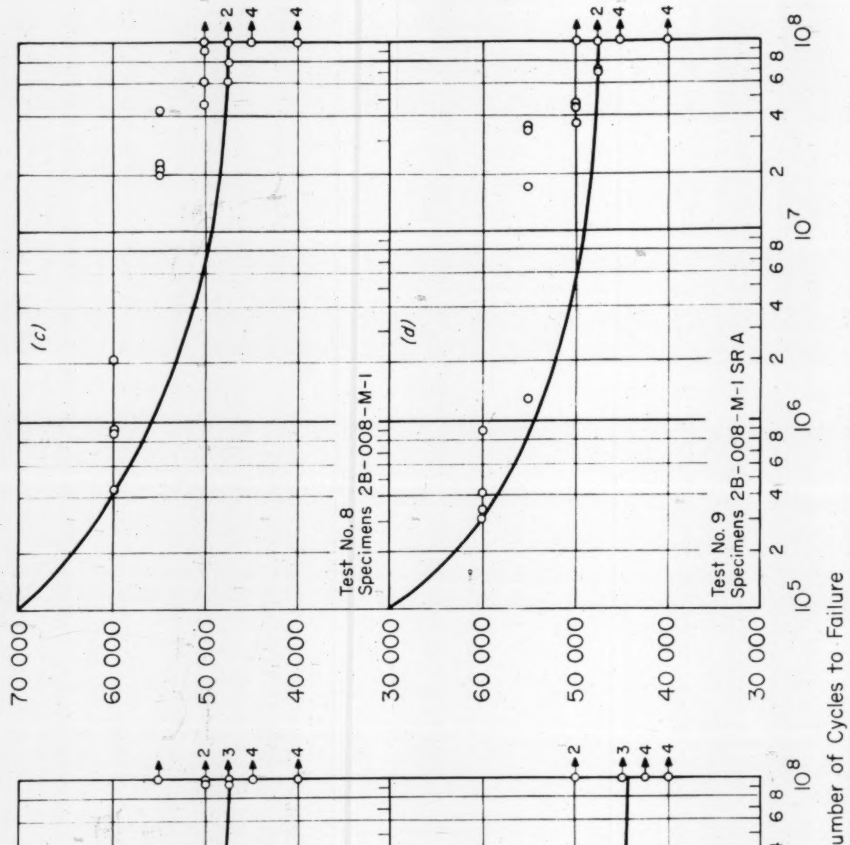


FIG. 3.—Fatigue Characteristics of Hard-Drawn Alloy A Phosphor Bronze Rod Showing Effect of Polishing and Stress-Relief Annealing.

R. R. Moore specimens. Diameter = 0.300 in. Nominal ready-to-finish grain size = 0.008 mm.

DISCUSSION OF DATA

A study of the fatigue data presented in Figs. 1, 2, and 3 shows that the minimum fatigue strength of hard-drawn phosphor bronze rod at 10^8 cycles of reversed bending varies considerably for unnotched rotating-beam specimens obtained from various sources. These data are summarized in Table I.

TABLE I.—SUMMARY OF TEST PROGRAM AND FATIGUE DATA.

Test	Specimen Description					Fatigue Data Shown in Figure:	Minimum Fatigue Strength at 10^8 Cycles of Reversed Stress, psi
	Surface	Nominal Grain Size, mm	Average Surface Roughness, microinches	Designation	Prepared by:		
No. 1	Machined and polished	0.035	3.5	1A-035-MP-1	Machine shop A	1(*a*)	44 000
No. 2	Machined and polished	0.035	2.0	1B-035-MP-1	Machine shop B	1(*b*)	35 000
No. 3	Machined and polished	0.008	2.5	2B-008-MP-1	Machine shop B	2(*a*)	37 000
No. 4	Machined	0.008	10	2C-008-M-1	Research laboratory C	2(*b*)	46 000
No. 5	Machined and polished	0.008	3.5	2D-008-MP-1	Fatigue testing laboratory D	2(*c*)	50 000
No. 6	Machined and polished	0.008	1.0	2B-008-MP-2	Machine shop B	3(*a*)	47 500
No. 7	Machined, polished, and stress-relief annealed[a]	0.008	1.0	2B-008-MP-2-SRA	Machine shop B	3(*b*)	44 500
No. 8	Machined	0.008	17	2B-008-M-1	Machine shop B	3(*c*)	47 500
No. 9	Machined and stress-relief annealed[a]	0.008	17	2B-008-M-1 SRA	Machine shop B	3(*d*)	47 500

[a] 4 hr at 400 F in hydrogen.

From the data in Table I it can be seen that the estimated minimum fatigue strength at 10^8 cycles of reversed stress for the machined and polished specimens made from the 0.035-mm hard-drawn alloy A phosphor bronze rod was approximately 25 per cent higher for specimens obtained from machine shop A than for those obtained from machine shop B, even though both lots of specimens were prepared in accordance with the information given in the manual, and despite the fact that specimens from the latter shop had a better surface finish.

In the case of the 0.008-mm material, it should be noted that the machined but unpolished specimens from laboratory C, which were used in test No. 4, had a minimum fatigue strength approximately 25 per cent higher than that of the machined and polished specimens prepared by machine shop B. That is, the smoother surface finish of the polished specimens used in test No. 3 did not seem to improve the fatigue life of the phosphor bronze rod. On the other hand, machined and polished specimens prepared by laboratory D, which were used in test No. 5, had a minimum fatigue strength of 50,000 psi, which is not appreciably different from the 46,000 psi observed in test No. 4 for machined but unpolished specimens prepared by laboratory C.

In this case, the specimens with the smoother surface finish (test No. 5) had a fatigue strength slightly but not significantly higher than that of the rougher machined but unpolished specimens used in test No. 4.

The second lot of machined and polished specimens prepared by machine shop B from the 0.008-mm rod (test No. 6) had a minimum fatigue strength that differed markedly from the fatigue strength of the first lot (test No. 3), the values for the minimum fatigue strength being approximately 30 per cent higher for the second lot. The results for the second lot of polished specimens (test No. 6) prepared by machine shop B were comparable to those previously obtained in fatigue tests on machined but unpolished specimens prepared by laboratory C (test No. 4) and machined and polished specimens of the same material prepared by laboratory D (test No. 5).

The fatigue strength for the same group of polished specimens after stress-relief annealing (test No. 7) was slightly lower than for the unannealed specimens. From this, it may be assumed that the polishing operation left slight residual compressive stresses on the surface which tended to raise the fatigue strength. Attempts to verify this assumption by removing metal from one side of the broken fatigue specimen and observing the change in curvature indicated that the residual stresses, if present, were quite low and were compressive.

Unpolished specimens prepared by machine shop B (test No. 8) had a minimum fatigue strength at 10^8 cycles comparable to the values observed for similar unpolished specimens previously prepared by laboratory C. In this case, stress-relief annealing the fatigue specimens prior to test did not seem to affect the test results, thus indicating the absence of any significant residual stresses.

Specimens of widely different surface roughness (1.0 to 17 microinches) had approximately the same fatigue life. This is evident from a comparison of test Nos. 4, 5, 6, and 8. Hence, variations in surface roughness appear to have less effect on fatigue life than variations due to residual stresses resulting from the different polishing operations.

CONCLUSIONS

1. The information given in the ASTM Manual on Fatigue Testing is not sufficiently explicit to enable the experimenter to obtain reproducible test results on specimens of the same strain-hardenable material prepared by different machine shops or by the same shop at different times. This lack of explicit instructions makes it difficult, if not impossible, to compare fatigue results published in the literature unless the effect of specimen preparation is also given for each lot of material tested. This is seldom included in technical articles except for investigations such as those cited in references 2 through 18.

2. More consistent agreement was obtained for specimens of hard drawn phosphor bronze rod machined to final size than for machined and polished specimens as called for in the manual.

3. Inasmuch as stress-relief annealing did not affect the fatigue results for specimens of the hard-drawn phosphor bronze rod that were machined and not polished, it may be concluded that these specimens were relatively free from residual stresses.

4. In the phosphor bronze rod used in these studies there appears to be little or no correlation between the minimum fatigue strength at 10^8 cycles and surface roughness for average surface roughness ranging from 1.0 to 17 microinches.

5. Tests made on rotating-beam fatigue specimens prepared from hard-drawn phosphor bronze rod in accordance with the information given in the manual

are more likely to evaluate variations in residual stresses induced in the preparation of test specimens than differences between materials.

6. The primary source of variation in the fatigue test results obtained on R. R. Moore type specimens made from strain-hardenable material such as phosphor bronze rod appears to be associated with the polishing operation rather than with the machining or the final surface roughness. Therefore, the polishing operation should either be eliminated or detailed more explicitly.

7. Round-robin tests should be made by ASTM Committee E-9 on Fatigue to permit the development of instructions on specimen preparation that will insure the accomplishment of the stated objectives, namely, the correlation of test results between laboratories and the obtaining of comparable and reproducible test results.

Acknowledgment:

The authors wish to express their appreciation to Miss E. Kerner for preparing the illustrations.

REFERENCES

(1) Manual on Fatigue Testing, Section IV, "Specimens and Their Preparation," Am. Soc. Testing Mats., (1949). (Issued as separate publication *ASTM STP No. 91.*)

(2) R. L. Ferguson, "Effect of Surface Finish on Fatigue Properties at Elevated Temperatures," *NACA RM-E51-D17*, Nat. Advisory Committee for Aeronautics, June 26, 1951.

(3) R. L. Ferguson, "A Further Investigation of the Effect of Surface Finish on Fatigue Properties at Elevated Temperatures," *NACA Technical Note 3142,* Nat. Advisory Committee for Aeronautics, March, 1954.

(4) R. J. Love, "The Influence of Surface Condition on the Fatigue Strength of Steel," in Properties of Metallic Surfaces, *Monograph and Report*, Series 13, Institute of Metals, p. 161.

(5) M. J. Brown, "Effects of Finishing on 75S-T6 Aluminum Alloy," *Product Engineering*, Vol. 25, Sept., 1954, p. 163.

(6) R. C. Miller, Jr., and A. W. Brunot, "Fatigue Tests of Steel Specimens Prepared for Metallizing," *Welding Journal*, Vol. 33, June, 1954, p. 275.

(7) G. Panseri and F. Gatto, "Fatigue Resistance Test of Light Alloys—Influence of Experimental Factors," *Aluminio*, Vol. 24, No. 5, Sept.–Oct., 1955, p. 447 (*in Italian*).

(8) F. H. Vitovec and H. F. Binder, "Effects of Specimen Preparation on Fatigue," WADC *Technical Report 56-289* Wright Aero. Development Center (ASTIA Document No. AD97211), Aug., 1956.

(9) G. M. Sinclair, H. T. Corten, and T. J. Dolan, "Effect of Surface Finish on the Fatigue Strength of Titanium Alloys RC-130B and Ti 140A," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 79, Jan., 1957, p. 89.

(10) J. L. Waisman, "Factors Affecting Fatigue Strength," in Metal Fatigue, McGraw Hill Book Co., Inc. New York, N. Y. (1959).

(11) R. A. Baughman, "Effect of Hardness, Surface Finish, and Grain Size on Rolling Contact Fatigue Life of M50 Bearing Steel," ASME-ASLE Lubrication Conference (to be published in Transactions, Am. Soc. Mechanical Engrs. (1960).

(12) P. G. Fluck, "The Influence of Surface Roughness on the Fatigue Life and Scatter of Test Results of Two Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 584, (1951).

(13) P. Gimmel, "Effect of Surface Condition on Fatigue Bending Strength," *Werkstoffe und Korrosion*, Vol. 2, Dec., 1951, p. 461 (*in German*).

(14) A. T. Steer, "Electromachining and Superfinishing as a Production Tool, II. The Finishing of Highly Stressed Parts," *Electroplating and Metal Finishing*, Vol. 8, July, 1955, p. 245.

(15) B. Cina, "The Effect of Surface Finish on Fatigue," *Metallurgia*, Vol. 55, Jan. 1957, p. 11.

(16) L. P. Tarasov and H. J. Grover, "Effects of Grinding and Other Finishing Processes on the Fatigue Strength of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 668 (1950).

(17) D. N. Cledwyn-Davies, "Effect of Grind-

ing on the Fatigue Strength of Steels," *Engineer*, Vol. 198, Aug. 20, 1954, p. 270; also *Proceedings*, Inst. of Mechanical Engrs., Vol. 169, No. 2, p. 83 (1955).

(18) L. P. Tarasov, W. S. Hyler, and H. R. Letner, "Effects of Grinding Direction and of Abrasive Tumbling on the Fatigue Limi of Hardened Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 528 (1958).

(19) G. R. Gohn and W. G. Bader, "The Tensil Properties of Hard Drawn Alloy A Phosphor Bronze Rod," see p. 843 of this publication.

DISCUSSION

MR. JOHN N. KENYON[1] (*presented in written form*).—In December, 1958, Mr. A. J. Kennedy of the Aeronautical Institute in Cranfield, England, published an article entitled "Fatigue Since Wöhler." He said that no plausible theory of fatigue failure had been advanced in the past century; that Wöhler himself could return and feel at home in any laboratory. Mr. Kennedy attributed the lag in creative endeavor to the fact that investigators were slaves to the *S-N* diagram and, inferentially, to the machined specimen. This inference is based on the fact that upward of 90 per cent of fatigue studies today are with the rotating-beam machine.

Pursuant to the above, Mr. Gohn is performing a service in pointing out the shortcomings in the machined specimen and the potential dangers of accepting misleading data. Too often the young research worker starts out with a theoretical concept and, by way of verification, carries out a few fatigue tests by a standardized technique. He obtains wide scatter data, employs statistical rules to eliminate the isolated points, and confirms his theory in the qualitative sense. It is our experience that wide scatter in data is more often due to lack of sensitivity in testing technique.

Dealing with the fatigue properties of wire material for years, this investigator has long since departed from the machined specimen and tests wire in the processed condition. Surface condition is admittedly a controlling factor but not always what one might expect. For instance, the roughing of a surface may actually raise the fatigue strength.

The accompanying Fig. 4 shows in the lower curve the approximate fatigue strength of steel wire in the processed condition. The middle curve shows the fatigue strength of the wire after drawing through a diamond dust die while rotating. The upper curve shows the increased fatigue strength obtained by drawing twice through the diamond dust die, wherein the directions of rotation were reversed. This wire had the appearance of a rat-tail file under the microscope. The increase of fatigue strength is explainable by the fact that the fine diamond scratching hardened the surface; the scratching is probably akin to shot-peening.

Figure 5 shows in the lowest of the group of three similar curves the fatigue strength of stock wire. The next higher curve shows the fatigue strength obtained by etching for $3\frac{1}{2}$ hr with 10 per cent hydrochloric acid. The top curve was obtained on wire that was drawn twice through the diamond dust die with directions of rotation reversed plus 5 hr etching in 10 per cent hydrochloric acid. The gain in fatigue strength over the stock wire is on the order of 20 per cent. These gains are often lost when the surface-treated wires are tested in corrosive environment. Note the data from tests in distilled water.

These data bring out forcibly the fact that the preparation of fatigue specimens

---

[1] Fatigue of Materials Laboratory, Princeton, N. J.

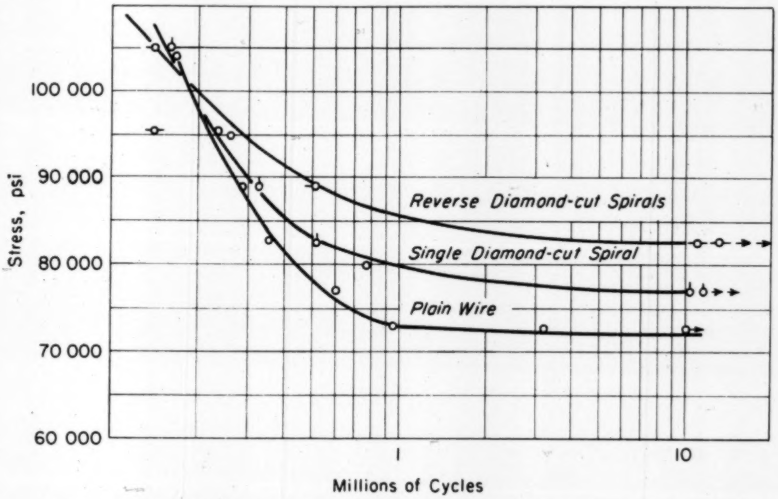


FIG. 4.—Fatigue Test Results for Cable Grade Steel Wire (0.038 in. Diameter).

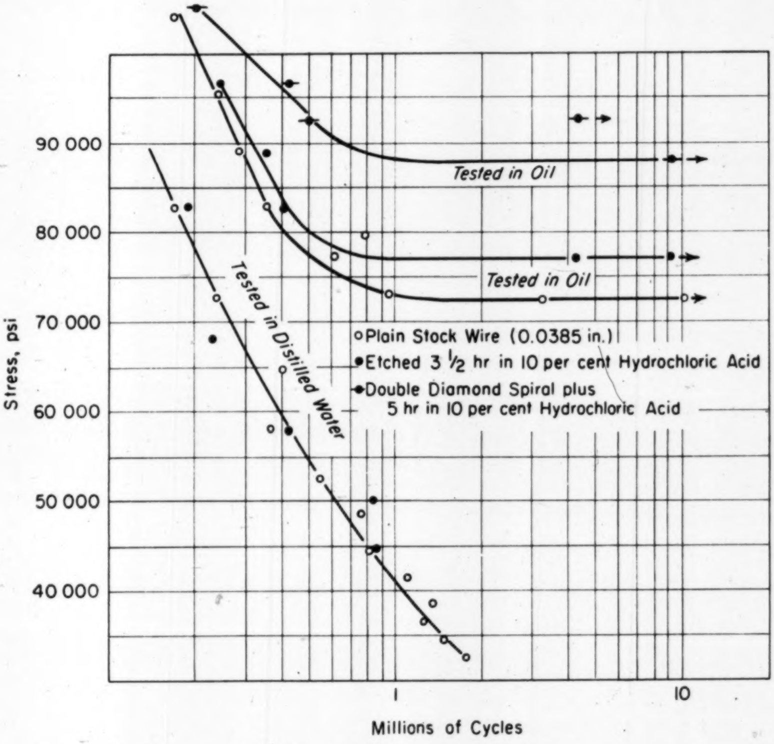


FIG. 5.—Fatigue Test Results for Cable Grade Steel Wire (0.0385 in. Diameter), Tested in Oil and in Distilled Water.

must be given more objective consideration. The authors' findings should have far-reaching implications.

MR. G. R. GOHN (*author*).—There are a few comments that I would like to add to Mr. Kenyon's discussion. Our paper gives pertinent references to the literature which deal with the effect of different surface treatments on fatigue life (see references (**2**) to (**18**), incl, of the paper). The work of Vitovec and Binder (reference (**8**) of the paper) nicely summarizes studies of this type which were made primarily to evaluate the effect of various surface finishes on fatigue life rather than the effect of chance variations in specimen preparation.

As Mr. Kenyon points out, the use of machined fatigue test specimens yields test data which have certain shortcomings. For example, if the machined specimens do not simulate the conditions observed in service, correlation between laboratory and field tests becomes difficult. When the material is used in the finished product without machining (for example, wire or flat springs punched from strip) tests such as Mr. Kenyon advocates on the material as processed will yield the most useful data. Even when both the specimen and the finished parts are machined, differences in the results obtained in the two tests will be observed if the methods of processing the two and the size and shape of the parts are not comparable. Nevertheless, there is a definite field for the use of a standardized unnotched machined test specimen in the evaluation of the fatigue characteristics of materials. It is also essential in obtaining a datum point for any study on the effect of notches or other stress concentration on fatigue.

MR. E. H. SCHUETTE.[2]—This paper critically analyzes a specific set of instructions for specimen preparation rather than the fatigue test itself. In that connection, I find nothing to criticize in the results obtained, nor are they especially surprising. This appears to be a carefully conducted program, and it should stand as a warning to anyone who might labor under the delusion that the fatigue strength of a material is some single-valued and easily reproducible quantity.

The conclusions reached, with the exception of Nos. 5 and 7, seem sound enough. However, I must take issue with those two conclusions, and also with the strong underlying implication in all the conclusions that the ASTM Manual on Fatigue Testing (reference (**1**) of the paper) has been proved inadequate.

Conclusion 5 states that tests in accordance with the ASTM Manual on Fatigue Testing "are more likely to evaluate variations in residual stresses. . . than differences between materials." I cannot agree that they will evaluate either one. They are certainly likely to be influenced by residual stresses, but only a meticulously planned and conducted program like the one in this paper can offer a real evaluation.

Conclusion 7 asks for a round-robin program to permit writing of more detailed standards on specimen preparation. I am convinced we can use our investigative time, talent, and equipment to better purposes than this. We already have in the literature a good bit of evidence that standardized preparation will not necessarily assure identical results, and also that the influence of preparation may be quite different for different materials, and therefore require different standards. Added to these considerations is the impossibility of writing instructions that will mean the same thing to all people.

Thus, I, for one, consider the proposed program an impossible one to fulfill. I also consider it an undesirable one. I was not a party to the preparation of the

---

[2] 4313 Sherwood Court, Midland, Mich.

ASTM Manual on Fatigue Testing, but some limited inquiries lead me to the conclusion that its original authors intended it as a guide, containing sufficient detail to call attention to some of the many precautions that must be observed if one is to conduct reproducible fatigue tests. It is not in any sense—nor do I think it purports to be—a standard.

I would, in fact, offer strenuous objections to any attempts to make it a standard. I like my freedom to adjust methods to suit the case at hand. I see little utility in a knowledge of the "true fatigue strength" of a material, except for comparison with the fatigue strength under some revised condition which should be simultaneously evaluated if the comparison is to have any meaning. To have much utility in predicting serviceability, both specimen preparation and the environment should be adjusted to match as nearly as possible the conditions of service.

I would not have a detailed and arbitrary standard for specimen preparation. By the time we know enough to prepare such a standard intelligently, we will no longer have any use for the test so standardized.

Perhaps the language of the ASTM Manual on Fatigue Testing should be revised in some respects to make it clear that it is not a standard, and that the researcher should take it only as an indication of necessary precautions.

MR. GOHN.—Mr. Schuette appears to be in general agreement with the conclusions set forth in our paper. However, his disagreement with conclusions 5 and 7 warrants further consideration.

If we correctly interpret his objections to the use of the information given in Section IV of the ASTM Manual on Fatigue Testing as the basis for preparing fatigue test specimens and the use of data obtained from tests on such specimens in comparing the fatigue properties of materials, we find that in theory, we are in agreement. Unfortunately fact and theory differ. In recent years, under the impetus of government sponsored research, many universities, government agencies, and commercial testing laboratories have undertaken fatigue studies. Many of the investigators engaged in this work have no background in fatigue. They purchase an ASTM Manual, read it and believe that it represents the last word in fatigue testing. The fact that no revision has been made in the Manual on Fatigue Testing since it was first issued in 1949, except to issue a new section on definitions[3] and a supplement dealing with the statistical analysis of fatigue data (*ASTM STP 91-A*), fails to deter them from following the suggestions outlined in the Manual as the basis for specimen preparation and fatigue testing. Having done this, they expect to obtain results which will be comparable and reproducible and which can be correlated with those of other laboratories. This paper shows that such confidence is not justified.

On the basis of the work cited in our paper it was felt that the process finally used in preparing the machined and polished specimens for test No. 6 was sufficiently under control so that specimens could be prepared which would give reproducible results. Accordingly, a third lot (designated 2B-008-MP-3) of machined and polished specimens were obtained from machine shop B. At the same time a group of machined specimens (designated 2B-008-M-2) and a group of ground specimens (designated 2B-008-G-1) were also obtained from machine shop B. Half of the machined specimens were electropolished after first mechanically polishing until all tool marks were removed on a muslin buffing wheel impregnated with rouge paste and rotated at 3450 rpm. Electropolishing was carried out in an 85 per cent orthophosphoric acid solution in which the specimen was rotated on its own axis until a total of 0.005 in. of metal had been removed. This resulted in the removal of any residual stresses arising from the machining and mechanical polishing operations. All four lots of specimens were tested in a "staircase test" to determine the mean fatigue strength at 10^8 cycles of reversed stress. The results, as determined from the data presented in Fig. 6, were:

---

[3] See Appendix to 1959 Report of ASTM Committee E-9 on Fatigue, *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 596 (1959).

Specimen Preparation	Mean Fatigue Strength at 10^8 Cycles of Reversed Stress, psi
Machined	48 800
Machined and mechanically polished	51 200
Machined and electropolished	47 200
Ground	52 500

Since the staircase test gives fatigue strengths in terms of the mean rather than the minimum values used by the authors in the paper, the results of the two types of tests cannot be directly compared when runouts are obtained. Nevertheless the close agreement between the mean values obtained in the staircase test and the median values of fatigue strength which can be estimated from the S-N curves shown in Figs. 2 and 3 of the paper for specimens prepared in the same manner indicates that reproducible results can be obtained.

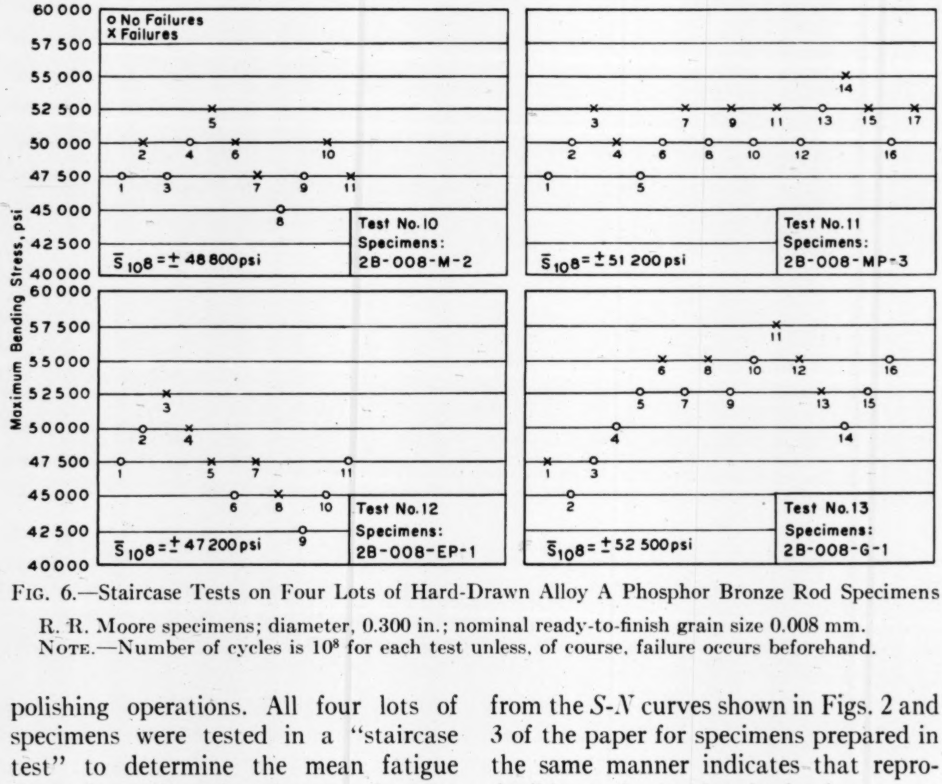


FIG. 6.—Staircase Tests on Four Lots of Hard-Drawn Alloy A Phosphor Bronze Rod Specimens

R. R. Moore specimens; diameter, 0.300 in.; nominal ready-to-finish grain size 0.008 mm.
NOTE.—Number of cycles is 10^8 for each test unless, of course, failure occurs beforehand.

The lower results obtained on electropolished specimens is in line with the findings of Vitovec and Binder (reference (8) of the paper) and the increased fatigue strength of the ground specimens is in line with the data in the literature (references (15) and (17) of the paper). Yet all of these methods of specimen preparation (except electropolishing) are in line with the suggestions in the Manual on Fatigue Testing.

Exception is taken to Mr. Schuette's criticism of conclusion 7 of the paper. Without standardization of specimen preparation and test methods, a comparison of fatigue data as reported in the literature becomes meaningless. Nor are the difficulties as insurmountable as he infers. As we see it, the problem is no different than that posed by the development and standardization of the Rockwell hardness tester. Because this test did not measure hardness in terms of the fundamental units of length, mass, and time, the National Bureau of Standards refused to calibrate and standardize the tester. Therefore a group participated in a round-robin test from which the use of standard hardness blocks was developed. By the use of such blocks any tester can be checked and adjusted so that comparable hardness values can be obtained on the same material regardless of where or on what Rockwell machine the test is performed. A similar undertaking on the part of ASTM Committee E-9 on Fatigue could perform the same function for those interested in fatigue testing. We are pleased to report that the chairman of Committee E-9 has appointed a Task Group on Specifications for Specimen Preparation to initiate this work. Whether their recommendations are incorporated in the Manual on Fatigue Testing or are issued as a Recommended Practice is immaterial. Either would be a step forward.

MR. DAVID E. MARTIN.[4]—I think it is possible to outline two recommended procedures and find that one of the procedures is very sensitive to human error. That is, you can have a given man perform the same procedure on separate specimens at two different times and get different results.

I think that a set of standards might take this into consideration, and we ought to develop a procedure that is relatively insensitive to human error. Apparently the ASTM Manual on Fatigue Testing does not provide such a procedure. I believe what Mr. Gohn has recommended is very valid, and that we should go ahead and try to develop a recommended procedure that is insensitive to this problem of human error.

MR. GOHN.—We are pleased to find support for our suggestions in Mr. Martin's comments.

MR. J. VIGLIONE (*by letter*).[5]—The title of this paper is very misleading, since it appears from the title that the paper is concerned primarily with a critical evaluation of the R. R. Moore rotating beam fatigue machine itself. Such is not the case. The paper deals solely with the effects of specimen surface finish upon the fatigue life of hard-drawn phosphor bronze rod. It would seem that this paper would have a greater value if the title were more appropriately descriptive of the nature of the investigational work done, particularly for ease and convenience in future listings in the Annual Fatigue Reference and the ten-year bibliography.

The writer agrees with the authors that Section IV (Specimen and Preparation) of the ASTM Manual on Fatigue Testing is inadequate to serve as a satisfactory standard. No effort has been made to standardize on one best method of polishing specimens; the manual includes three, and makes the very important observation that "Procedures for preparing that portion of the test specimen on which calculated fatigue stresses are imposed require standardization to permit correlation of results between laboratories and to obtain comparable and reproducible results."

---

[4] Senior Research Engineer, Special Problems Department, Research Laboratories, General Motors Corp., Warren, Mich.

[5] Aeronautical Materials Laboratory, Naval Air Material Center, U. S. Naval Base, Philadelphia, Pa.

In all fairness, it must be pointed out that this manual was not intended to be a standard. Rather, its stated purpose is to "supply information to those setting up new laboratory facilities," and to set up "recommended practices which may later on be crystallized into standards."

It is agreed by now that a standard on specimen preparation should be established to ensure uniformity in this respect, to the end that reproducible results may be obtained and findings between laboratories may be correlated. The writer agrees with the authors that ASTM Committee E-9 on Fatigue, a group well qualified to do so, should make every effort to develop and establish this standard for specimen preparation as soon as possible.

The authors have performed an invaluable service in so ably calling attention to the need for establishing a standard on specimen preparation, and are to be complimented for this.

In view of the authors' stated objective to demonstrate that the manual's instructions on specimen preparation are inadequate and that polishing may have a great effect on the fatigue life of certain materials, it is unfortunate that they have not seen fit to include in this paper the description of the polishing procedure used in this investigation to compare polishing effects. The paper states that the shops and laboratories were requested to finish the specimens by machining and polishing in accordance with the information given in Fig. 9 and Section IV of the manual, and since three polishing procedures are listed therein, the question arises as to whether the same or different polishing procedures were used. It cannot be determined from the paper whether test groups made by different machine shops used the same or different polishing procedures, and whether test groups made by the same commercial machine shops at different times used the same or different polishing procedures.

The writer takes exception to the authors' statement in the synopsis that "the rotating-beam fatigue test becomes a measure of the effect of residual stresses resulting from the methods used in preparing the test specimens—particularly the polishing operation—rather than a determination of the fatigue properties of the material." At the Naval Air Material Center, considerable work has been done with rotating-beam fatigue machines, and correlated fatigue data have been accumulated over a period of years. With a local standard uncomplicated polishing procedure, an excellent degree of reproducibility in fatigue life has been obtained for aluminum and steel alloys despite the fact that some cases involved different heats of the same steel, different time intervals, different fatigue machines, and different personnel machining, polishing, and testing specimens. Experience has shown that complicated polishing procedures are no more effective in obtaining fatigue values that are in agreement, than is a uniform standard surface finish not necessarily highly polished.

MR. R. R. MOORE (*by letter*).[6]—This paper is an interesting contribution to that elusive and much investigated problem of surface finish of fatigue specimens. Reliable and comparative endurance limit values for materials cannot be obtained unless methods of preparation of test specimens are such as not to induce extraneous effects.

This leads directly to the question as to whether the ASTM Manual on Fatigue Testing should be criticized for not being a satisfactory standard for preparation of fatigue test specimens. It is quite apparent that it is too general to serve as a standard. When it was written it was not intended to be a standard, as witnessed by the statement on page 1 of the introduction which states "The purpose of the manual is to *supply information* to those setting up new laboratory facilities, to *aid* in properly operating the equipment, and to *offer advice* in presentation and interpretation of the data" (italics added). Therefore, in the absence of specific and sufficient instructions it would be expected that considerable variation in finished surfaces would result, as has been ably shown by the authors.

---

[6] Chief Metallurgist, Naval Air Engineering Facility, U. S. Naval Base, Philadelphia, Pa.

Nevertheless, there should be an effort made by some competent body to write a process specification or at least a recommended practice. I agree with the author that this would be a worthwhile task for Committee E-9, and one which it is well qualified to perform.

The authors have presented data to show that it is possible to produce compressive stresses in the surface of certain bronze specimens during the polishing operation. This is a matter of considerable importance and should be pursued further to determine whether other materials are similarly affected, to what extent, and how this can be overcome. In view of the fact that polishing was indicated to have a very important effect and that the Manual on Fatigue Testing describes several methods, it is regrettable that the authors have not indicated specifically which method was followed in each case.

Upon reading the title of this paper I drew the conclusion that it was concerned only with the type of this particular test or the performance of the testing machine. Of course upon reading the entire paper it was clearly evident that the paper only concerned preparation of test specimens, and the test data obtained are not only applicable to the R. R. Moore machine and specimen, but also, at least qualitatively, to cantilever rotating-beam specimens such as have been used by Krouse, McAdam, and others. In the second paragraph of their paper the authors quote instructions from the Fatigue Manual for preparation of test specimens as being specifically applicable to the R. R. Moore test. I find that these instructions are listed under a general heading "Cylindrical Specimens," and therefore the instructions are equally applicable to the cantilever rotating-beam specimen. Interestingly enough, the most elaborate details in the manual for polishing specimens are quoted from methods adopted at the Naval Engineering Station and the University of Illinois, neither of which used the R. R. Moore test.

It is suggested that the value of this paper would be greatly enhanced as a reference work if the title reflected the fact that the paper is concerned with the preparation and surface finish of fatigue specimens.

MESSRS. G. R. GOHN AND A. FOX (*authors' closure*).—The objection raised by Messrs. Viglione and Moore to the misleading title have, we believe, been met by the revision in the subtitle. Both commentators agree that the Manual on Fatigue Testing was not intended to be a standard for fatigue testing but merely a guide. However, since it has been used as such, both concur with the authors that a standard on specimen preparation should be established to the end that reproducible results may be obtained and findings between laboratories may be correlated.

In answer to Mr. Viglione, the exact details of the preparation of the test specimens used in the first few tests are not known. At present, the specimens are all obtained from machine shop B. Those which are machined and mechanically polished as suggested by the Manual are machined at a speed of approximately 500 rpm and a feed of 0.0015 in. The machined specimens are then mounted so that they revolve about their own axis while driven through a variable speed transmission. A $\frac{1}{2}$-in. wide fiber glass tape, impregnated with 600-mesh silicon carbide, is drawn over a rubber wheel of approximately 4 in. in diameter mounted at right angles to the axis of the specimen. This rubber wheel is moved back and forth across the surface of the specimen in the arc of a circle of approximately 30 in. diameter. The impregnated tape is moved forward at a speed of 1 ft per min, thus presenting a fresh cutting surface to the specimen about every third stroke. By driving the specimen at a variable speed a crosshatched polishing takes place, thus tending to eliminate the scratches of the previous stroke. In the final polishing operation, a soft plastic tape impregnated with 600-mesh silicon carbide is substituted for the fiber glass tape while the speed of specimen rotation is adjusted to give essentially a longitudinal polish.

We agree with Mr. Moore that the conclusions drawn from our tests on rotating-beam fatigue specimens are, at least qualitatively, applicable not only to the R. R. Moore type of specimen but also the cantilever type of rotating-beam specimens such as those used by Krouse, McAdam, and others.

Mr. Viglione indicates that his experience at the Naval Air Material Center in the use of the polishing technique outlined in the first column of page 35 of the Manual on Fatigue Testing has proved effective in obtaining reproducible fatigue test results. This leads the authors to hope that standardization is not as insurmountable as some of the commentators have indicated and would argue well for the success of the Task Group on Standardization of Specimen Preparation in completing the assignment given it by ASTM Committee E-9.

The authors are indebted to those who have participated in both the oral and written discussion of the paper in emphasizing the significance of these studies.

ADDITIONAL STUDIES OF EFFECTS OF ANODIC COATINGS ON THE FATIGUE STRENGTH OF ALUMINUM ALLOYS*

BY G. W. STICKLEY[1]

SYNOPSIS

As a supplement to an earlier paper (1),[2] the effects of some surface treatments and of additional anodic coatings, some as thick as 0.002 in., on the fatigue strength of 7075-T6 aluminum alloy were determined. The results confirmed that thin anodic coatings have no significant effect, but showed that thick coatings, not sealed, decrease the fatigue strength considerably. The preliminary surface treatments that were used had no effect, but sealing caused decreases. In the presence of severe stress raisers such as mechanical notches, thick coatings caused no further decrease in fatigue strength.

Included also are the results of tests of sealed anodically coated alclad sheet of aluminum alloys 2024-T3, 7075-T6, and 7178-T6. The fatigue strengths of such sheet with thick coatings generally were lower than those of alclad-type sheet without anodic coating.

The anodic treatment of aluminum alloys has been used commercially for many years for various purposes. In some earlier applications, the main object was to provide improved resistance to corrosion, and in those cases, the coatings were relatively thin (0.0001 to 0.0002 in.). While coatings continue to be used for this purpose, there are many applications where other qualities are of primary importance. In more recent years, there has been considerable use of thicker coatings (0.0005 to 0.002 in.) where hardness and resistance to abrasion are very important. In certain other applications, such as the thinner coatings on alclad sheet, the advantage of anodic coatings is in their ability to be impregnated with dyes and pigments, in order to obtain desired colors.

In considering the effects of anodic coatings on fatigue strength, it may also be important to consider the preparation, if any, of the surface for anodizing. If the surface is sufficiently smooth and clean, no preparation is needed; but if the surface is not smooth and clean enough, a chemical treatment may be used that in itself may have an effect on fatigue strength.

In an earlier paper by the author, in 1950 (1),[2] the effects of some anodic coatings on the fatigue strength of several aluminum alloys were reported representing a total of seven combinations of five coatings on four alloys. The thicknesses of these coatings generally were 0.0005 in. or less. This paper showed that, for the coatings and alloys tested, these anodic coatings had little, if any, effect on the fatigue strengths of the

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Assistant Chief, Mechanical Testing Division, Alcoa Research Laboratories, New Kensington, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—COMPOSITIONS OF MATERIALS TESTED.

Alloy	Composition, per cent				
	Copper	Manganese	Magnesium	Zinc	Chromium
2024[a]	4.5	0.6	1.5	...	...
7075[b]	1.6	...	2.5	5.6	0.3
7178[b]	2.0	...	2.7	6.8	0.3

[a] The cladding of Alclad 2024 sheet is 99.5 per cent aluminum.

[b] The cladding of Alclad 7075 and Alclad 7178 sheet is aluminum alloy containing 1.0 per cent zinc.

TABLE II.—TENSILE PROPERTIES OF MATERIALS TESTED.

Alloy and Temper	Form	Size, in.	Direction	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent
7075-T6	rolled-and drawn rod	¾	longitudinal	86 400	74 700	14.5
	extruded rod	¾	longitudinal	89 200	80 800	9.0
Alclad 2024-T3[a]	sheet	0.063	longitudinal	66 100	48 400	19.2
			transverse	65 200	43 800	19.2
Alclad 7075-T6[a]	sheet	0.063	longitudinal	75 600	68 200	11.0
			transverse	75 500	65 600	10.8
Alclad 7178-T6[a]	sheet	0.063	longitudinal	83 700	78 000	11.8
			transverse	84 400	74 100	11.5

[a] The nominal thickness of the coating on each surface of this thickness of Alclad 2024 and Alclad 7075 sheet is 2½ per cent of the total thickness. For Alclad 7178 it is 4 per cent.

alloys, but that coatings as thick as 0.0010 in. seemed to have slight detrimental effects at higher stresses. In general, these results were in agreement with work done by others, as reviewed in that paper.

In the meantime the effects of thicker coatings have been reported by several investigators. Johnson (2) reported tests of several wrought aluminum alloys which showed that coatings reduced the fatigue strength at 10^7 cycles in amounts from about 20 to 60 per cent, and that for coating thicknesses from 0.001 to 0.005 in. on sheet of several alloys, the decreases were independent of thickness. .On alclad-type sheet, Johnson found that anodic coatings decreased the fatigue strength beyond the decrease associated with the alclad coating. On alloy castings, however, there was no reduction, since the effects of the pores and irregularities in the surface of a casting were more harmful than the effects of the coating. Wernick and Pinner and others (**3,4**) found that a 0.002-in. coating decreased the fatigue strength of 0.190-in. 7075-T6 sheet at 10^5 cycles about 40 per cent.

The additional work described in the present paper includes studies of the effects of the following on fatigue strength: (*a*) chemical preparation treatments on rod of 7075-T6 alloy; (*b*) anodic coatings, in thicknesses ranging from 0.0001 to 0.002 in., on rod of 7075-T6 alloy; (*c*) sealing of coatings on rod of 7075-T6 alloy; and (*d*) anodic coatings on alclad sheet of alloys 2024-T3, 7075-T6, and 7178-T6.

MATERIAL AND COATINGS

The first group of tests was made using ¾-in. 7075-T6 rod, most of the tests being made on a single lot of rolled-and-drawn rod and the remainder on one lot of extruded rod. The second

TABLE III.—PREPARATION OF SURFACES.

	Test Group	Preliminary Preparation of Surface[a]	Anodic Treatment				Sealing Treatment	
			Alumilite	Nominal Thickness of Coating, in.	Current, amp per sq ft	Time, min	Time, min	Medium[b]
Tests of Rod			NO TREATMENT					
	A-1	none	...	...	...	...	...	...
			PRELIMINARY TREATMENT ONLY					
	A-2	R1	...	...	...	...	...	...
	A-3	R5	...	...	...	...	...	...
			COATING SOLUTION: 10 PER CENT CrO_3 AT 95 F					
	B-1	none	...	0.0001	...	65	none	...
	C-1	none	...	0.0002	...	305		
	D-1	none	...	0.0004	4.1	160		
			COATING SOLUTION: 15 PER CENT H_2SO_4 AT 70 F					
	E-1	none	...	0.0001	12	6	none	...
	E-2	R1					none	...
	E-3	R5					none	...
	E-4	none					10	BW
	F-1	none	No. 204	0.0005	12	30	none	...
	F-4	none					30	BW
	G-1	none	...	0.001	12	66	none	...
	G-4	none					10	BW
	G-5	none					15	D
	H-1	none	...	0.002	24	60	none	...
	H-2	R1						
	H-3	R5						
			COATING SOLUTION: 12 PER CENT H_2SO_4 PLUS 1 PER CENT OXALIC ACID, AT 50 F					
	J-1	none	No. 225	0.001	36	20	none	...
	J-4	none					10	BW
	K-1	none	No. 226	0.002	36	40	none	...
Tests of Sheet			COATING SOLUTION: 15 PER CENT H_2SO_4 AT 70 F					
	L-1	R1	No. 201, gold 145	0.0003	...	15	10	BW
	M-1	R1	No. 215, gold 145	0.001	...	54	10	BW

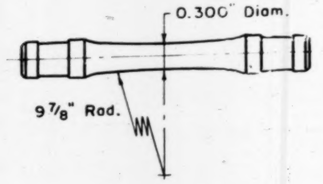
---

[a] R1 consisted of etching in 5 per cent NaOH for 30 sec at 150 F.
R5 was a brightening dip for 90 sec at 190 F.

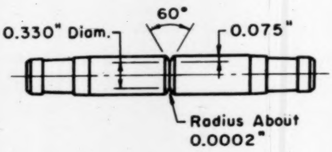
[b] BW boiling water; D, dichromate.

group of tests was made on 0.063-in. Alclad 2024-T3, Alclad 7075-T6 and Alclad 7178-T6 sheet.

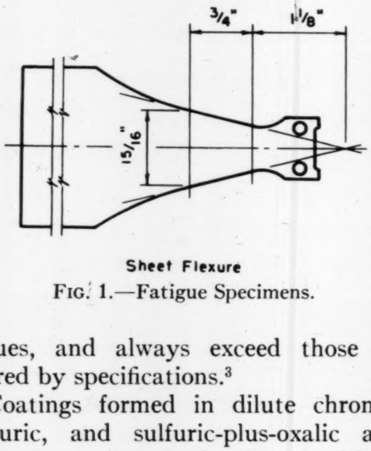
The compositions of the various alloys are shown in Table I. The tensile properties of the materials tested, shown in Table II, generally agree with the typical values, and always exceed those required by specifications.[3]



Rotating-Beam, Smooth



Rotating-Beam, Notched



Sheet Flexure

FIG. 1.—Fatigue Specimens.

Coatings formed in dilute chromic, sulfuric, and sulfuric-plus-oxalic acid electrolytes were used, with or without preliminary chemical treatments of the surface of the specimens, and with or without sealing. Some of the coatings were commercial, while others were experimental. Details concerning the various chemical treatments and coatings are summarized in Table III. The actual thicknesses of the anodic coatings agreed closely with the nominal values.

Of the two chemical treatments, R1 is an etch with caustic, which roughens the surface; and R5 is a chemical polishing treatment. The length of time of treatment generally depends on the condition of the original surface, and accordingly was relatively short in these tests.

EQUIPMENT AND METHODS OF TEST

The fatigue tests of rod were made in R. R. Moore rotating-simple-beam machines and those of sheet were made in Sonntag SF-2 repeated-flexure machines (5). The specimens are shown in Fig. 1. In general, smooth specimens were used, although in two groups notched specimens also were tested. The procedures used in machining the various specimens were the same as those described by the author in an earlier paper (1), except that the specimens from the extruded rod, which were machined with tracer equipment and had a surface finish of less than 20 rms, were not polished. The anodic coatings were applied after the machining and any polishing of the test sections were completed.

Since anodic coatings are prone to craze at high stresses, and since this might affect the fatigue properties of an

---

[3] Tentative Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211 – 59 T), 1959 Supplement to Book of ASTM Standards, Part 2, p. 216.

Tentative Specification for Aluminum-Alloy Extruded Bars, Rods, and Shapes (B 221 – 59 T), 1959 Supplement to Book of ASTM Standards, Part 2.

Tentative Specification for Aluminum-Alloy Sheet and Plate (B 209 – 59 T), 1959 Supplement to Book of ASTM Standards, Part 2, p. 188.

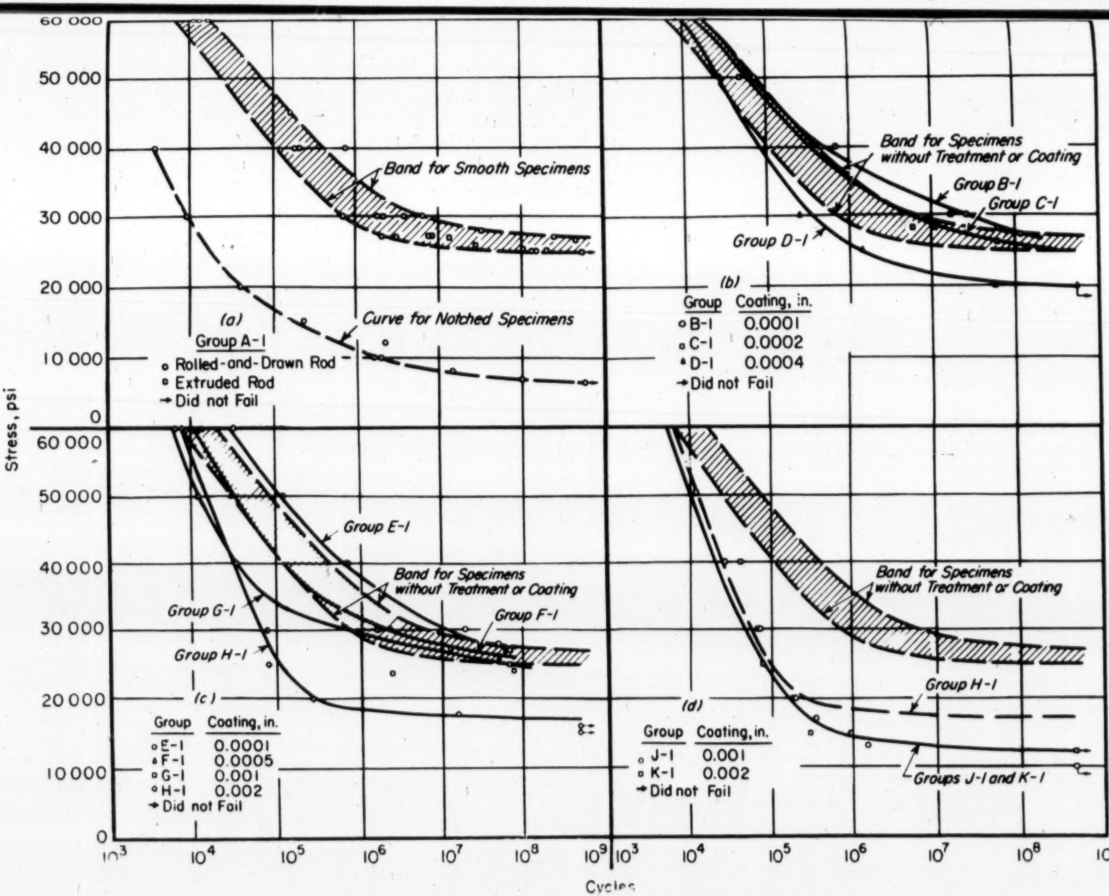


FIG. 2.—Rotating-Beam Fatigue Data for 7075-T6 Rod.

(*a*) Without surface treatment or coatings (smooth and notched specimens).
(*b*) Coated by the chromic-acid anodic process, not sealed (smooth specimens; no prior treatment).
(*c*) Coated by the sulfuric-acid anodic process, not sealed (smooth specimens; no prior treatment).
(*d*) Coated by the sulfuric-plus-oxalic acid anodic process, not sealed (smooth specimens; no prior treatment).

alloy, some observations were made during static tensile and bending tests and during fatigue tests of the alclad sheet, to determine the tensile stresses at which crazing first occurred. This was done by making Faxfilm replicas of the coated surfaces of specimens during loading, and noting if and when crazing occurred. This was followed by metallographic examination.

DISCUSSION OF TESTS OF ROD

The results of the tests of smooth and notched specimens without any surface treatment or coating (group A-1) are shown in Fig. 2(*a*). They show that the fatigue strength of the extruded rod, which was used only in certain tests of smooth specimens, was about equal to that of the rolled-and-drawn rod. As a basis for comparison, the band for smooth specimens and the average curve for notched specimens are shown in subsequent plots.

The series of tests of smooth specimens with coatings applied without preliminary surface treatments will be discussed first, then those with preliminary surface treatments, and finally those with sealed coatings.

In the tests of specimens coated by the chromic-acid process (groups B-1, C-1, and D-1) no prior surface treatments were used, and the coatings were not sealed. As can be seen in Fig. 2(*b*), the effects of the 0.0001- and 0.0002-in. coatings were not harmful, and in some cases were slightly beneficial. The effect of the 0.0004-in. coating, which is thicker than it would be practical to obtain commercially with the chromic-acid anodic process, was harmful at fatigue stresses of 20,000 to 40,000 psi, the decrease in fatigue strength at 10^8 cycles being about 25 per cent.

The results of the four groups of tests of specimens coated by the sulfuric-acid process; without prior surface treatment and not sealed, are shown in Fig. 2(*c*) (groups E-1, F-1, G-1, and H-1). Again, the 0.0001-in. coating was not harmful. When the 0.0005-in. coating was used, the fatigue strengths were not as high as with the thinner coating, but still were within the band for the original material. The 0.001-in. coating generally had a harmful effect at stresses above 30,000 psi. The 0.002-in. coating was definitely harmful at all stresses, the decrease in fatigue strength at 10^8 cycles being about 35 per cent.

The results of the two series of tests using 0.001- and 0.002-in. coatings made in a sulfuric-plus-oxalic acid electrolyte, without prior surface treatment and not sealed (groups J-1 and K-1), are shown in Fig. 2(*d*), together with the curve shown previously in Fig. 2(*c*) for the 0.002-in. coating made in a sulfuric-acid electrolyte (group H-1). Both of the coatings made in sulfuric-plus-oxalic acid had a considerably harmful effect, and the fatigue strengths were the same whether the thickness was 0.001- or 0.002-in. At stresses below 20,000 psi the fatigue strengths were lower than for the 0.002-in. coating applied in a sulfuric acid electrolyte (group H-1), the reduction in fatigue strength at 10^8 cycles being about 55 per cent.

The effects of the two chemical treatments, sometimes used prior to anodic treatments, are shown in Fig. 3(*a*). Neither the R5 bright dip alone (group A-3) nor the R1 etch alone (group A-2) had any significant effect, although it will be noted that the fatigue strength was slightly lower with the R1 treatment than with the R5. It is possible, of course, that a longer and more severe treatment of the R1 type could have a harmful effect.

When a 0.0001-in. coating was applied with sulfuric-acid electrolyte after either of these prior treatments (groups E-2 and E-3), there was no significant differ-

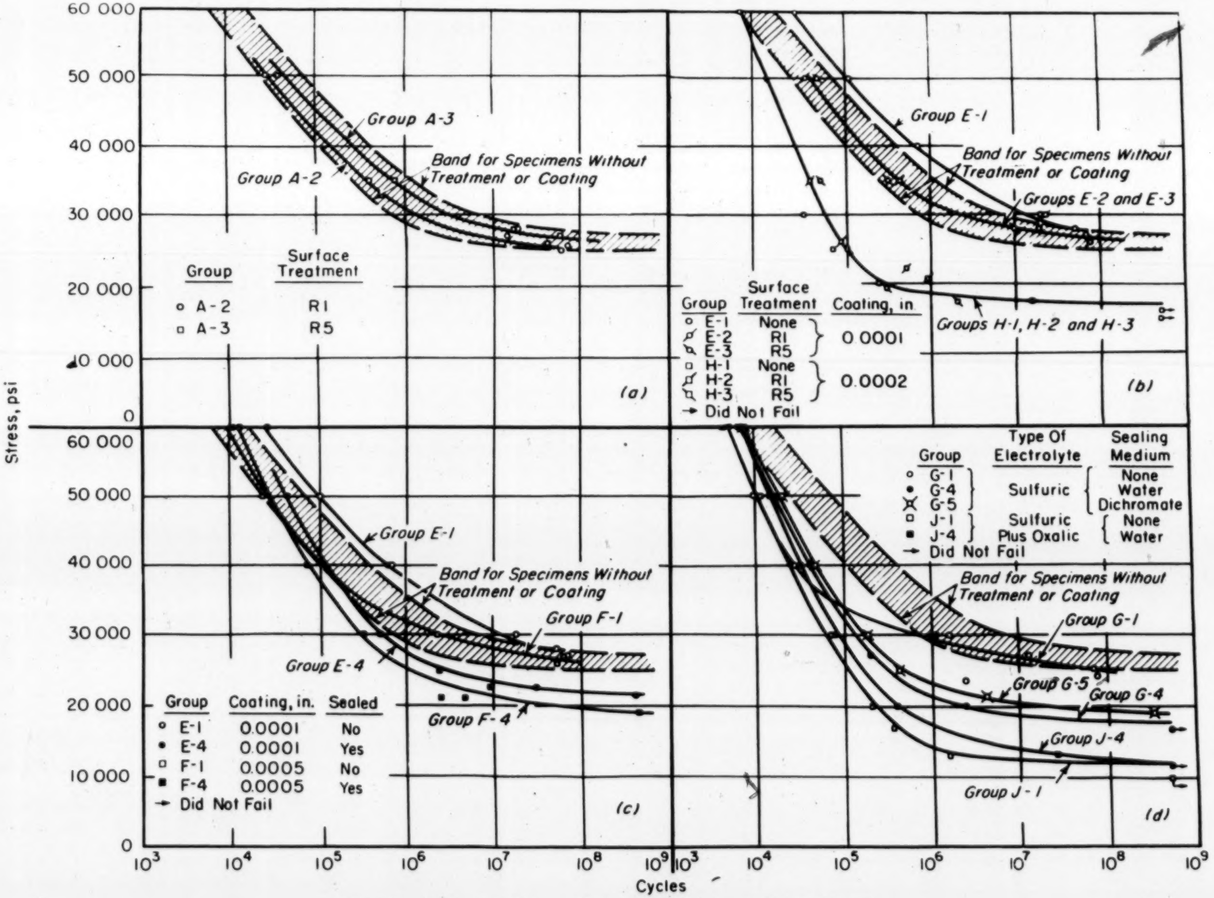


FIG. 3.—Rotating-Beam Fatigue Data for 7075-T6 Rod.

(*a*) With chemical surface treatments (smooth specimens).
(*b*) Coated by the sulfuric-acid anodic process, with and without prior chemical treatments, not sealed (smooth specimens).
(*c*) With sealed anodic coatings made by the sulfuric-acid process, without prior treatment (smooth specimens).
(*d*) With sealed 0.001-in. coatings made without prior treatment (smooth specimens).

ence whether the R1 or R5 treatment had been used, as can be seen in Fig. 3(*b*). Furthermore, since most of the data points lie within the band, there was no significant effect on fatigue strength. Similarly, when these preliminary treatments were used before applying a 0.002-in. coating with the same electrolyte (groups H-2 and H-3), the effect was the same as when no preliminary treatment (group H-1) was used.

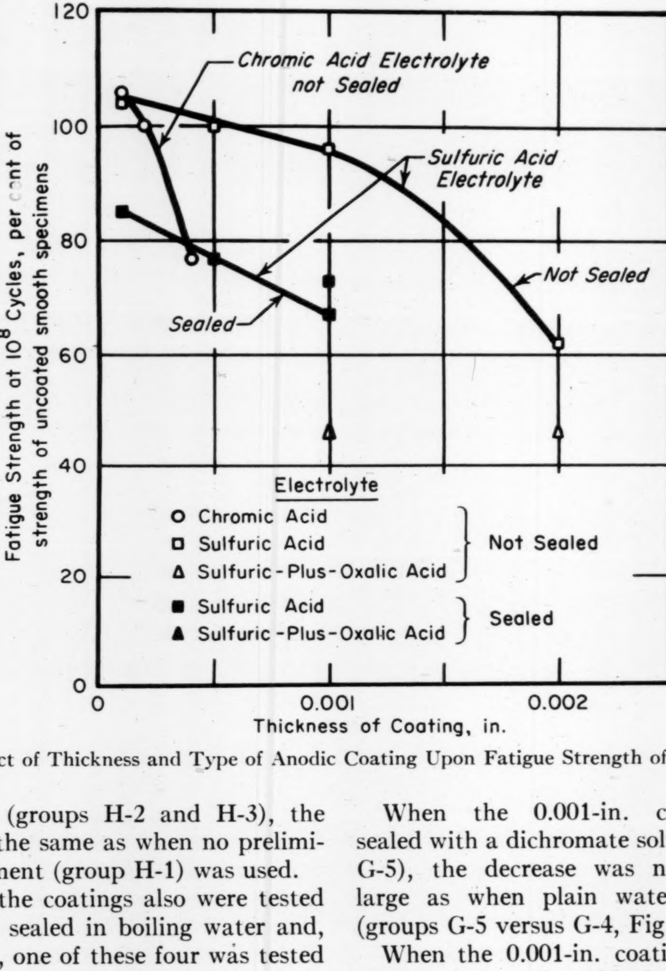


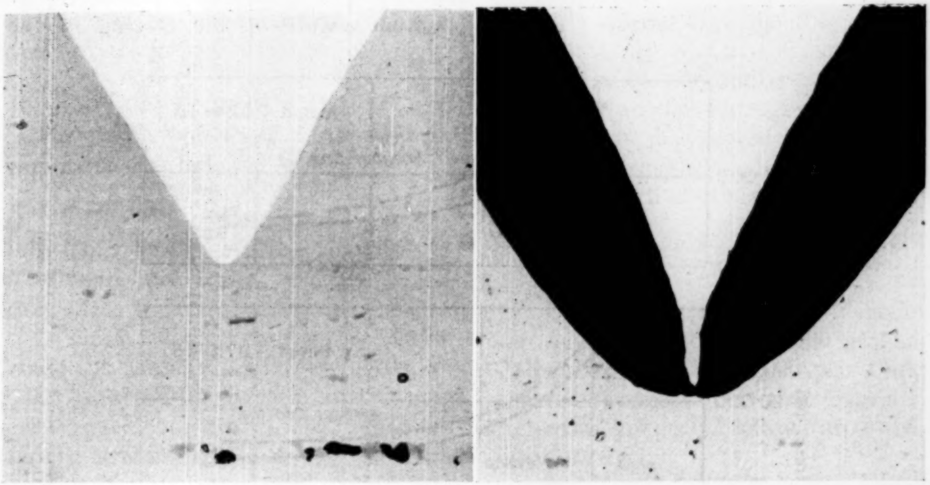
FIG. 4.—Effect of Thickness and Type of Anodic Coating Upon Fatigue Strength of 7075-T6 Rod.

Four of the coatings also were tested after being sealed in boiling water and, in addition, one of these four was tested after being sealed with a dichromate solution. The data for the 0.0001-, 0.0005-, and 0.001-in. coatings made in sulfuric-acid electrolyte and sealed in boiling water (groups E-4, F-4, and G-4), plotted in Figs. 3(*c*) and (*d*) together with the corresponding data for the unsealed coatings (groups E-1, F-1, and G-1), show that the sealing decreased the fatigue strengths of each. The decreases in fatigue strength at 10^8 cycles, from sealing alone, were from 20 to 30 per cent.

When the 0.001-in. coating was sealed with a dichromate solution (group G-5), the decrease was not quite as large as when plain water was used (groups G-5 versus G-4, Fig. 3(*d*)).

When the 0.001-in. coating made in sulfuric-plus-oxalic acid electrolyte was sealed with water (group J-4), however, the sealing seemed to improve the fatigue strength slightly, although the net decrease still was considerable.

The effects of the various coatings upon the fatigue strength at 10^8 cycles, as determined in tests of smooth specimens, are summarized in Fig. 4. The service involves simultaneous exposure to repeated stress and to corrosive conditions, the use of such coatings can be beneficial.



(*a*) As machined.

(*b*) With 0.002-in anodic coating made by the sulfuric-plus-oxalic acid process, not sealed.

FIG. 5.—Cross-sections of Notches in Fatigue Specimens of 7075-T6 Rod (×500, Reduced for Publication).

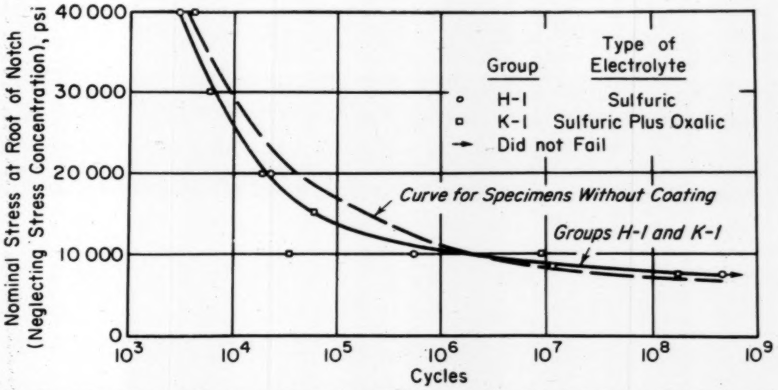


FIG. 6.—Rotating-Beam Fatigue Data for 7075-T6 Rod with 0.002-in. Coatings on Notched Specimens (no preliminary treatment; not sealed).

curves show graphically the relative harmful effects of thick coatings and of sealing, under the conditions used in the tests. It should be noted, however, as shown by experience, that when

The 0.002-in. coatings used in tests of smooth specimens in groups H-1 and K-1 were also used in tests of notched specimens. As can be seen in Fig. 5, the thickness of coating is not uniform, being very thin at the root. The anodic treatment, however, enlarged the root radius of the notch in the metal from 0.0002 in. to 0.0015 in. These coatings had no effect on notch fatigue strength at 10^8 cycles, although at stresses between 30,000 and 10,000 psi, the coatings appeared to have been harmful, as can be seen in Fig. 6.

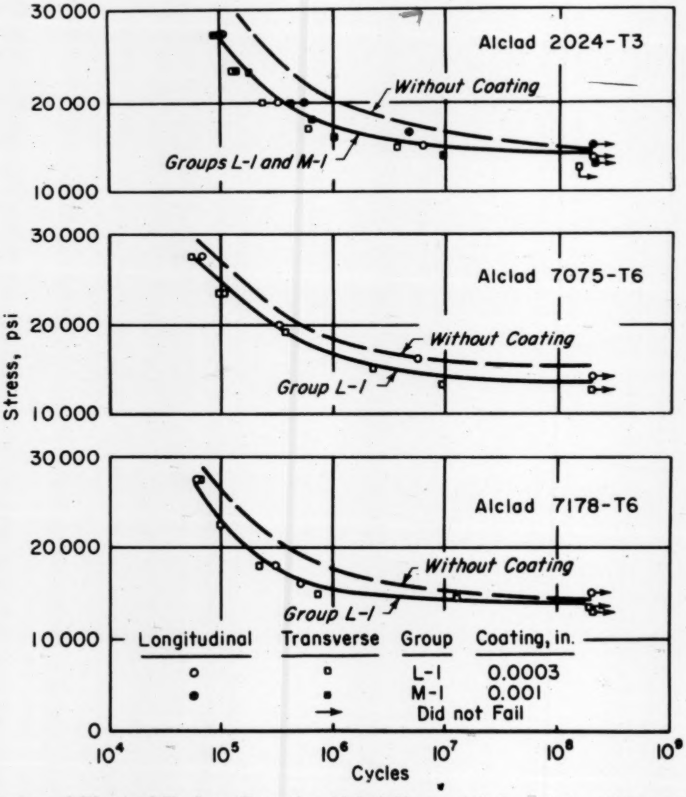


FIG. 7.—Reversed Flexural Fatigue Data for Alclad Sheet with Sealed Anodic Coatings Made by the Sulfuric-Acid Process, with Prior *R1* Etch.

DISCUSSION OF TESTS OF SHEET

In the static tensile and bending tests, crazing of the 0.0003-in. and 0.001-in. coatings was observed to begin at tensile stresses of 28,000 and 19,000 psi, respectively. In all fatigue tests, which were made at stresses as low as 12,500 psi, crazing occurred early in the fatigue test. The craze marks in the coating gave rise to the fatigue cracks within the metal itself. These cracks in the metal reproduced faithfully the straight, parallel nature of the crazing in the coating instead of having the feathery branched appearance that is typical of fatigue cracks in uncoated metal.

The results of the fatigue tests of the three alclad sheet alloys are plotted in Fig. 7, together with the original curves for the same respective lots of sheet without coatings. The results of all anodically coated specimens generally are below the curves for the same sheet without coatings, although the fatigue strengths of the Alclad 2024-T3 and Alclad 7178-T6 sheet at 10^8 cycles approached those of the original alclad sheet. In the tests of Alclad 2024-T3 sheet, there was no definite difference in effects of the two thicknesses of coatings (groups L-1 versus M-1).

SUMMARY

The results of these tests may be summarized as follows:

Rod of 7075-T6 Alloy:

1. Thin anodic coatings (0.0001- to 0.0002-in.), not sealed, had no significant effect on fatigue strength.

2. Thick anodic coatings (0.0004-in. made in chromic-acid electrolyte, or 0.001- to 0.002-in. made in sulfuric-acid electrolyte), not sealed, decreased the fatigue strength considerably. Under reversed bending stresses, the fatigue strength at 10^8 cycles was reduced as much as 35 per cent.

3. The 0.001- to 0.002-in. thick coatings made in sulfuric-plus-oxalic acid electrolyte and not sealed, decreased the fatigue strengths more than the same thickness of coatings made in sulfuric-acid electrolyte.

4. The R1 and R5 surface treatments, used alone or prior to anodic coating, had no significant effects on fatigue strength. It is possible, however, that a longer R1 treatment would have a harmful effect.

5. Sealing of anodic coatings in boiling water, regardless of their thickness, generally had a harmful effect on fatigue strength, which was in addition to any effect of the coating itself.

6. Thick anodic coatings did not add to the effects that severe stress concentrations, such as sharp notches, had upon fatigue strength at 10^8 cycles.

Sheet of Alclad 2024-T3, Alclad 7075-T6, and Alclad 7178-T6 Alloys:

7. Under static stress, 0.0003-in. anodic coatings crazed at 28,000 psi, and 0.001-in. coatings at 19,000 psi. Under repeated reversed stress they crazed at stresses as low as 12,500 psi after relatively few cycles.

8. Sealed anodic coatings 0.0003- to 0.001-in. thick reduced the fatigue strengths at small numbers of cycles, but generally had no effect on the fatigue strength at 10^8 cycles.

Acknowledgment:

The author is grateful to those members of the staff of the Alcoa Research Laboratories who conducted the tests and otherwise aided in preparing this paper.

REFERENCES

(1) G. W. Stickley and F. M. Howell, "Effects of Anodic Coatings on the Fatigue Strength of Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, pp. 735–743 (1950).

(2) Henry A. Johnson, "Mechanical Properties of Aluminum Hard Coatings," *Product Engineering*, Sept. 1954.

(3) S. Wernick and R. Pinner, "Surface Treatment and Finishing of Light Metals: Part II," *Sheet Metal Industries*, Vol. 32, pp. 345–356 (1955).

(4) Anonymous, "New Hard Coating Gains Wear Applications for Aluminum," *Materials and Methods*, Aug. 1950, p. 62.

(5) Manual on Fatigue Testing, Am. Soc. Testing Mats. (1949). (Issued as separate publication *ASTM STP No. 91*.)

DISCUSSION

Mr. Parks Toolin.[1]—The notch used was extremely acute. I am wondering if the author would like to comment on the effects of a less severe notch such as one might find in service.

Mr. G. W. Stickley (*author*).—We have not made any tests using anodic coatings on specimens with less severe notches. We would expect that there may be an effect, depending on the severity of the notch, which would be somewhere intermediate between the effects obtained for specimens with the severe notch and with no notch.

---

[1] Westinghouse Research Laboratories, Westinghouse Electric Corp., Pittsburgh, Pa.

DESIGN APPLICATIONS FOR IMPROVING FATIGUE RESISTANCE OF AIRPLANE STRUCTURES*

By C. R. Smith[1]

Synopsis

Methods for prolonging fatigue life of built-up structures are discussed. Lessening the load on the first rows of rivets in joints, providing interference fits, or a combination of both were found to be helpful.

Test data are presented showing that the ratios of effective spring constants of pressed-in bushings to those of surrounding structure can be used for predicting fatigue life. Effects of static overloading prior to testing are also discussed.

This paper is a continuation of the work presented at the Fatigue Session held in Los Angeles, Calif., in 1956 and published with Papers on Fatigue of Aircraft Structures (**1**).[2] The previous paper discussed rivet patterns for enhancing fatigue strength of joints. It stated that unequal load distribution among rivets, plus bending stresses, were contributing factors to short fatigue life, although no quantitative values were given for these effects. It further stated that stresses due to unequal distribution were superimposed on those due to bending, in the case of a lap joint, so that any effort to reduce either or both would go a long way toward solving the fatigue problem.

Mitchell and Rosenthal (**2**) have found that approximately twice as much load was carried by the end rivets of a four-rivet clevis joint as by the intermediate rivets. A visual demonstration of this distribution is given in Fig. 1. Here a piece of vinyl, cut to simulate a joint having four fasteners, is used as a photoelastic model. This simulates a joint made up of equal-thickness materials.[3]

Bending stresses at the first line of rivets are superimposed on axial stresses to further complicate the fatigue problem. Figure 2 shows a photoelastic pattern of a lap joint having three fasteners. Note the physical distortion caused by eccentric loading. This could be expected to continue until the centroidal axes of the two sheets become aligned, after which a decrease in the ratio of bending stress to axial stress could be expected. Accordingly, a joint such as this, when cycled at high load levels with a high mean stress, should exhibit a relatively better fatigue strength than at a lower load with zero mean stress. This point

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Design Specialist, Convair, Division of General Dynamics Corp., San Diego, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] This model and those shown through Fig. 4 were actually made to simulate spotwelded joints. However, the stress distribution should be more or less applicable to joints having mechanical fasteners. This work was done in connection with research on "Development of Elevated Temperature Sheet Spotwelded and Seam Welded Joints," Wright Air Development Division Contract No. AF 33 (616)-6293.

is particularly significant when two types of structure are compared by fatigue testing at the higher load levels to expedite testing. It does not necessarily follow that structures satisfactory here would be suitable for service loading.

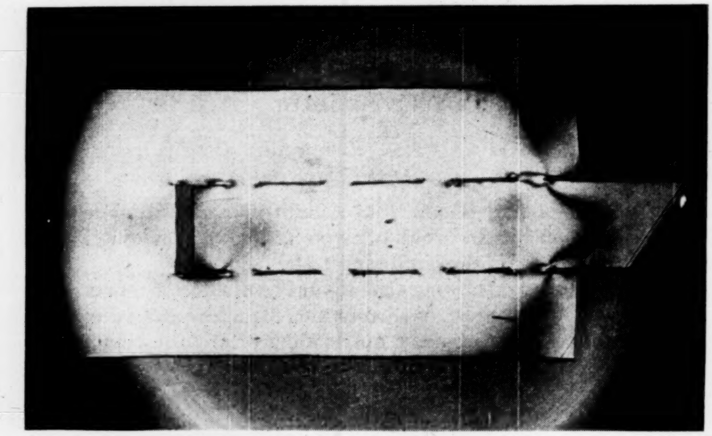


FIG. 1.—Photoelastic Model Showing Unequal Distribution of Load Among Fasteners in Clevis Joints.

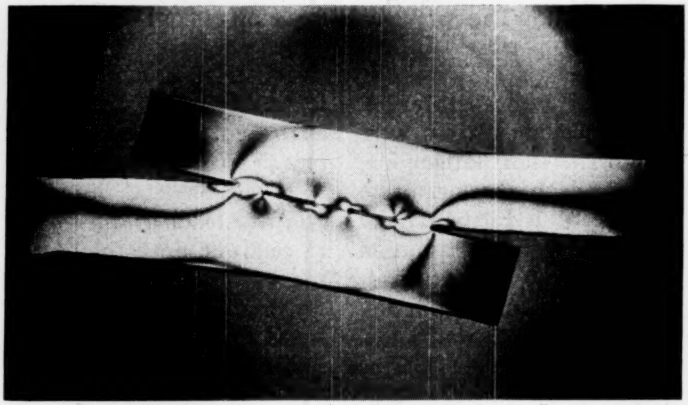


FIG. 2.—Photoelastic Model—Lap Joint—Effects of Bending at First Fastener.

Figures 3 and 4 show photoelastic patterns for tapered joints. Note that the load per fastener is more nearly equalized. Tapering joints as shown is not new, having been used for years for glued joints, and to a lesser extent in welded structures.

While the photoelastic experiments shown in Figs. 1, 2, 3, and 4 are only qualitative and somewhat simplified, they are extremely helpful in evaluating the existing problem and pointing out the direction for corrective measures. Designing a fatigue-resisting joint using mechanical fasteners presents a real problem, and some compromise between the existing and ideal scarfed joint is necessary. This is especially true in airplane structures where the need for an exterior smooth skin requires the use of flush fasteners, invariably resulting in a single-shear-type joint, which, as shown in Fig. 2, introduces bending to the problem of unequal load distribution. Stiffening the doubler for the purpose of eliminating bending exaggerates the unequal load distribution between fasteners. This can be minimized by inserting thin auxiliary doublers under the main splice so as to pick up an extra row of fasteners outside the main splice, or by driving auxiliary rivets through the edge of the doublers (3). The prime purpose here is to relieve either bending or axial stress at the first load-carrying fastener.

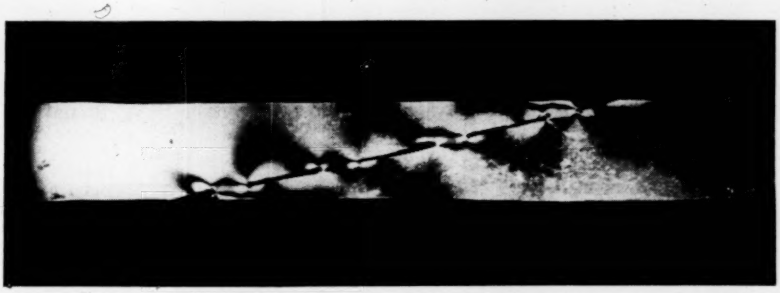


FIG. 3.—Photoelastic Model—Improved Stress Distribution in Plain Scarf Joint.

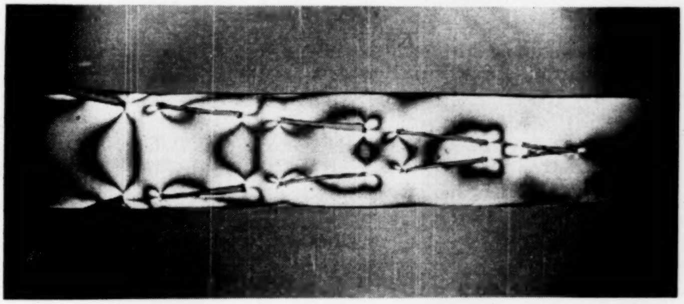


FIG. 4.—Photoelastic Model—Further Improvement in Double Scarf Joint.

TEST PROGRAM

In the previous paper (1) the authors showed that small amounts of static overloading were detrimental to the subsequent fatigue life of riveted joints, while high overloading improved life. Test data for one of these joints, a lap joint of 0.051-in.-thick clad 7075-T6 sheet, using $\frac{3}{16}$-in., 100-deg countersunk rivets spaced 1 in. apart in three rows $\frac{1}{2}$ in. apart, are given in Table I. It will be noted that where prestresses of 18,000 psi were applied prior to fatigue testing, a substantial reduction in life resulted, especially at the 10,000-psi and 5000-psi levels. Preloads of 25,000 psi caused some reduction in life, while preloads of 42,000 psi caused marked improvement in life. The loss and subsequent improvements were attributed to losses in propping[4] action afforded by the rivet and to gains in residual compressive stress and re-

---

[4] It was assumed that a tightly driven rivet would act as a prop which would prevent the return of stress to zero, even when unloaded. Slight amounts of overload would tend to loosen the rivet, so that it could not provide the support needed for this effect.

TABLE I.—EFFECT OF PRESTRESS ON LIFE OF RIVETED LAP JOINTS.[a]

Maximum Cyclic Stress (psi gross) $R = 0.1$	Maximum Static Prestress, psi			
	0	18 000	25 000	42 000
25 000	4 000 4 000 0.1077	... ...	... ...	20 000 20 000 0.1692
18 000	15 000 15 000 0.1068	... ...	13 000 13 000 0.0575	155 000 133 000 0.3010
15 000	36 000 34 000 0.1712	38 000 32 000 0.2720	127 000 81 000 0.512	812 000 748 000 0.2130
10 000	355 000 350 000 0.0812	102 000 98 000 0.1364	175 000 167 000 0.1494	10 169 000[b]
7 500	1 152 000 1 140 000 0.0944	... ...	... ...	... ...
5 000	6 752 000[b]	1 466 000 1 462 000 0.0316	4 940 000[b]	... ...
3 500	...	9 818 000[b]	...	...

[a] Values in this table are for arithmetic mean and log mean lives, and standard deviation, cycles. Standard deviations are with respect to log mean lives.

[b] One test point only.

TABLE II.—EFFECT OF PRESSED-IN BUSHINGS ON FATIGUE LIFE OF $\frac{3}{4}$-IN. THICK 7075-T6 LUGS HAVING $K_t = 3.5$ AND HOLE DIAMETER = 1.25 IN.

Bushing Wall Thickness, in.	Nominal Interference, in.	Interference per Inch of Hole Diameter, in.	Average Life[a] (4 specimens), cycles	Standard Deviation
No bushing	$1\frac{1}{4}$-in. diam bolt	...	34 500	0.095
$\frac{1}{8}$	...	...	29 250	0.165
$\frac{1}{8}$	0.005	0.004	255 250	0.284
$\frac{1}{8}$	0.0075	0.006	1 088 750	0.193
$\frac{1}{8}$	0.0075 (48 000 psi prestress)	0.006	541 250	0.403
$\frac{1}{4}$	0.005	0.004	316 750	0.269
$\frac{1}{4}$	0.0075	0.006	990 000	0.212
$\frac{1}{4}$	0.010	0.008	935 000	0.166

[a] Loaded at repeated 18,800-psi nominal net stress, $R = 0.015$. All tests made on Sonntag SF 10U axial loading fatigue testing machine.

distribution of load among rivets. The riveted joint in itself is too complicated to use as a vehicle to illustrate this mechanism. Accordingly, an attempt will be made here to analyze the same mechanism, using a simple lug with bushings pressed in at various amounts of interferences.

In Table II are presented data on the effect of pressed-in hardened steel bush-

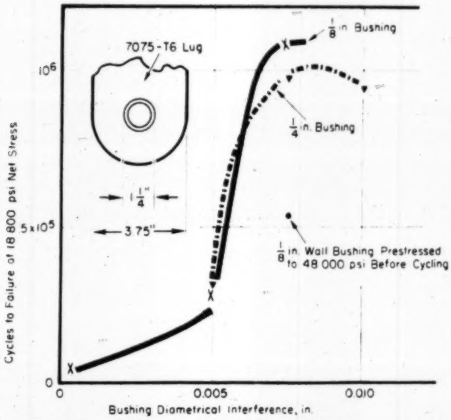


FIG. 5.—Effect of Bushing Interference on Fatigue Life of Lugs.

ings on fatigue life of a 7075-T6 lug 3.750 in. wide and 0.75 in. thick. The hole diameter was 1.250 in., giving a theoretical stress concentration of 3.5. Bushings of hardened steel having wall thicknesses of $\frac{1}{4}$ and $\frac{1}{8}$ in. were pressed in at varying amounts of interference, from zero to 0.010 in. Bushings were chamfered 10-deg to enable pressing in without extruding material from the lugs. The chamfered ends were ground off after pressing.

It will be noted that the average life to failure for four specimens having $\frac{1}{8}$-in. bushings but no interference was 29,250 cycles for repeated nominal net stresses of 18,800 psi. With 0.0075-in. interference, the average life was increased to 1,089,000 cycles. Average lives were less for either greater or lesser amounts of interference. These data are plotted in Fig. 5.

In Fig. 6 are *S-N* curves for smooth, axially loaded, 0.2-in. diameter specimens of 7075-T6 aluminum alloy as presented by Howell and Miller (4). In Fig. 7 are the same curves over which a line has been drawn to represent the locus of fatigue lives for varying stress ratios wherein a peak cyclic stress of 66,000 psi was maintained. In this case, 66,000 psi represents the nominal stress times the theoretical stress concentration factor for the lugs. Two test points are plotted on this line, one for the life of a lug with no interference and the other for a bushing interference of 0.0075 in.

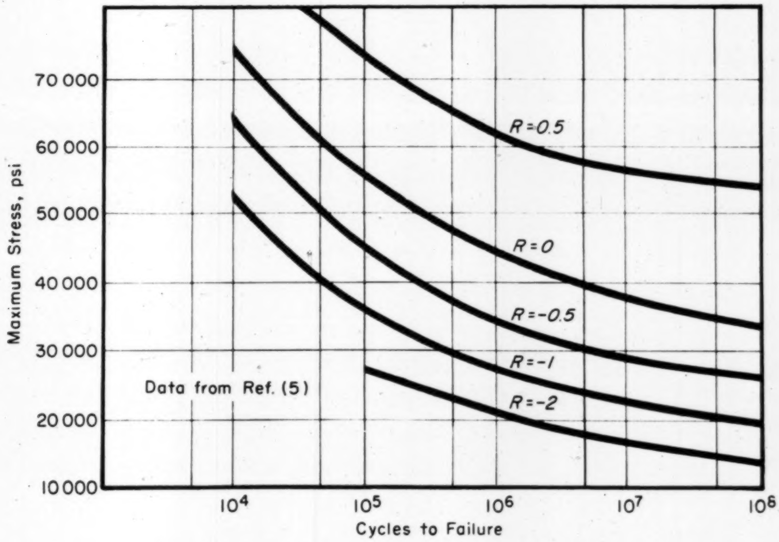


FIG. 6.—Direct-Stress *S-N* Fatigue Curves, 7075-T6.

Here, it can be seen that the fatigue life for the lug without interference falls about on the curve for $R = 0$ (actual test R was 0.015), indicating that the theoretical stress concentration factor of 3.5 was about right without further correction.[5] The point representing 0.0075-in. interference is open to question at this time, because it would appear that the interference should add to the peak stress. However, if we consider that the 0.0075-in. interference amounted to a nominal strain of 0.006 in. per in. (the hole in the lug was 1.25 in. in diameter) we find that an additional 0.0006 in. per in. strain would have been required to increase the maximum stress. It is assumed that the strain due to press fit at locations away from the concentration did not contribute to the fatigue damage.

According to the position of the point representing the lugs with 0.0075-in. interference (average for four specimens) and the relationship between the S-N curves for various stress ratios, it would appear that an effective stress ratio of about 0.6 would be in order. The term "effective" is used here because the lug behaved as though that were the stress ratio, despite the fact that actual loading was at a stress ratio of 0.015. Since the stress ratio is the minimum stress divided by the maximum stress, the minimum stress would amount to 66,000 × 0.6 = 40,000 psi. Assuming a modulus of elasticity of 10,000,000 psi for aluminum alloy, the strain for 40,000 psi would have amounted to 0.004 in. per in. In other words, the 0.0075-in. interference resulted in expanding the hole 0.004 in. per in., while the bushing shrunk 0.002 in. per in. (the 0.0075-in. interference amounted to 0.006-in. interference per inch of diameter).

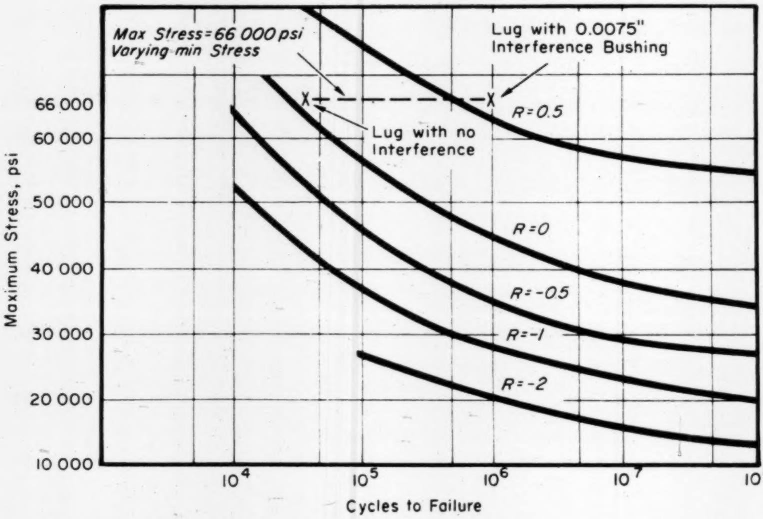


FIG. 7.—Effect of Interference Bushing on Fatigue Life of Lug, $K_t = 3.5$.

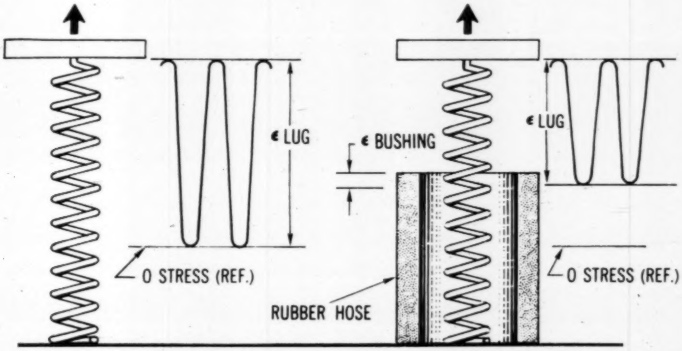
We can now say that the effective[6]

---

[5] Fatigue reduction factors or Neuber values are in common usage. It is possible that the bolt bending effect, which was not considered here, was enough to offset these effects.

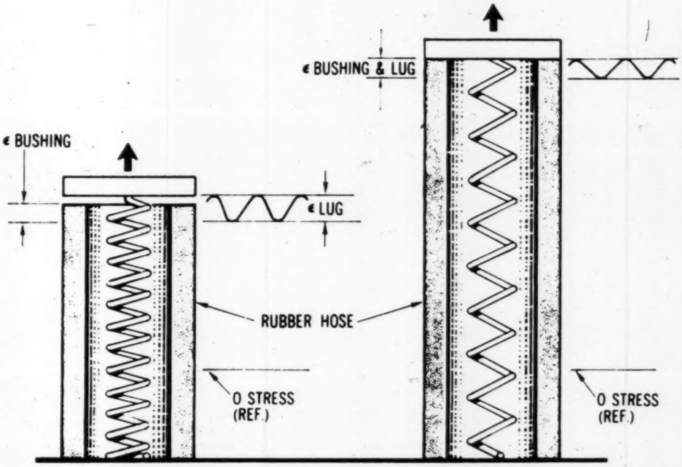
[6] The term "effective" is used here, since prorating the moduli of elasticity and areas of the bushing and lug would result in nothing like this ratio. This would lead one to believe that only the areas adjacent to the faying surface contributed much to the stiffness, that is, where the ratio of spring constants 3 to 1 instead of 2 to 1, this would be the case. Accordingly, it would seem that only a portion of the lug cross-sectional area was engaged in reacting the forces introduced by the bushing.

spring constant of the bushing was twice that of the lug material, and, knowing the relative spring constants, we can now predict fatigue life for any other amount of bushing interference. This can be best illustrated by a numerical example. However, before tackling this problem, a schematic diagram of what has been discussed should prove of assistance.



(*a*) Lug, plain hole, 34,500 cycles.

(*b*) Lug with bushing, low interference, 316,750 cycles.



(*c*) Lug with bushing, proper interference, 990,000 cycles.

(*d*) Lug with bushing, higher interference, 935,000 cycles.

FIG. 8.—Spring Analogy Depicting Bushing Interference.

A mechanism to explain these phenomena is best examined by an analogy. This time it has to do with springs. Remember, the assumption was made that the bushing acted as a prop, adding a floor, so to speak, under the stress pattern which prevented it from returning to zero.

In Fig. 8 are schematic sketches of a spring subjected to repeated tension loads. Figure 8(*a*) shows the sine curve generated by the displacement of an unrestrained spring for a given loading. The spring is used to simulate the most highly stressed inside fibers of a lug without a bushing. Figures 8(*b*), (*c*), and (*d*) show the spring subjected to the same loading, but restrained by props of varying heights, simulating bushings with different amounts of interference. The strain amplitudes in these sketches represent a movement of both lug and bushing. However, in Fig. 8(*b*) the bushing deformation is relatively small compared to that for the spring representing the lug. This simulates the case where only a small amount of interference is present.

Figure 8(*c*) shows a case where the interference was almost equal to the cyclic stress. The relative stiffness is evident from the small amplitude, which is representative of the combined spring constants of both lug and bushing. This represents the optimum amount of interference for this particular stress level. Greater amounts of interference such as that shown in Fig. 8(*d*) add to the peak stress, so that the fatigue life could be expected to be less than for that shown in Fig. 8(*c*),[7] although it could easily be greater than for that shown in Figs. 8(*a*) and (*b*) by virtue of the small cyclic amplitude.

One and only one amount of interference is optimum for a given stress amplitude. While that shown in Fig. 8(*c*) would be optimum for the amplitude shown, higher loading would benefit more by greater interference such as shown in Fig. 8(*d*), while for lower loading, the interference shown in Fig. 8(*b*) might be best.

An attempt will now be made to apply the above spring analogy directly to strain, using the experimental data from Table II for the necessary spring constants, and *S-N* data for smooth, axially loaded specimens for equivalent lives.

We have shown that an interference of 0.0075 in. would induce a residual stress in the lug of 40,000 psi. Assuming that the combination bushing and lug acts as a single spring and that the rate is constant from zero to 40,000 psi, were the bushing to be pressed in with an interference of 0.005 in., the residual stress would be $\frac{0.005}{0.0075} \times 40{,}000 =$ 26,600 psi. The new stress ratio would be $\frac{26{,}600}{66{,}000} = 0.4$, and the predicted fatigue life from the *S-N* curves in Fig. 6 would be about 300,000 cycles. This is in good agreement with the experimental average values of 316,750 for the $\frac{1}{4}$-in. thick bushings and 255,250 for the $\frac{1}{8}$-in. thick bushings.

It is of interest to note that little difference was found between fatigue lives for lugs with $\frac{1}{8}$-in. or $\frac{1}{4}$-in. thick bushings. This may be attributed to stiffness ratios being effective only in the faying surfaces of the journal, or to differences in bolt bending, bolts being 0.75 in. and 1 in. diameter for the $\frac{1}{4}$- and $\frac{1}{8}$-in. thick bushings, respectively.

So far, our discussion has covered only those conditions where interference did not add to the cyclic stress. In the case of the bushing having an 0.010-in. interference, some addition to the peak stress could be expected, because this would amount to an equivalent strain of 0.010/1.25, or 0.008 in. per in. This would be higher than the 0.0066 in. per in. peak cyclic strain assumed in these experiments.

An estimate of how much this would add to the peak stress can be obtained by adding the cyclic stress amplitude to the residual stress set up by interference. First, the residual stress would amount to twice that found for the 0.005-in. interference, or 2 × 26,600 = 53,400 psi. It is assumed that the spring rate is linear throughout these ranges. Secondly, the cyclic amplitude for the unbushed lug was found to be 66,000 psi at the point of stress concentration. However, the spring constant of the combined bushing and lug at this point is now three times[8] that of the unbushed lug, so the cyclic amplitude would be one-third as much, or 22,000 psi. Accordingly, the stress for 0.010-in. interference would amount to 53,400 psi residual stress plus 22,000 psi cyclic stress, giving a total of 75,400 psi. The corresponding stress ratio would be 53,400/75,400 = 0.71, and the predicted life from Fig. 6 would be about 700,000 cycles. The test average for the $\frac{1}{4}$-in. thick bushings was 935,000 cycles (average of four specimens).

---

[7] The cyclic amplitude, once the combined spring constants are fully employed, would be the same for any amount of interference greater than shown in Fig. 8(*c*). Photoelastic studies by Jessop, Snell, and Holister (**5**) lend support to this supposition.

Static preloading above the elastic limit introduces a problem that is relatively easy where no interference is present, but becomes very complex with interference. Especially pertinent are the compressive residual stresses acquired at the points of concentration. A method for determining the amount of residual stress, using a linear strain relationship in the plastic range, has been used by the author with satisfactory results for open notches (6). In essence, it assumes that a concentration is a geometric relationship and would be more appropriately expressed in terms of strain than stress. Accordingly, in order to find the stress at the point of concentration, one has only to multiply the nominal strain by the concentration for maximum strain, using stress values from ordinary stress-strain curves.

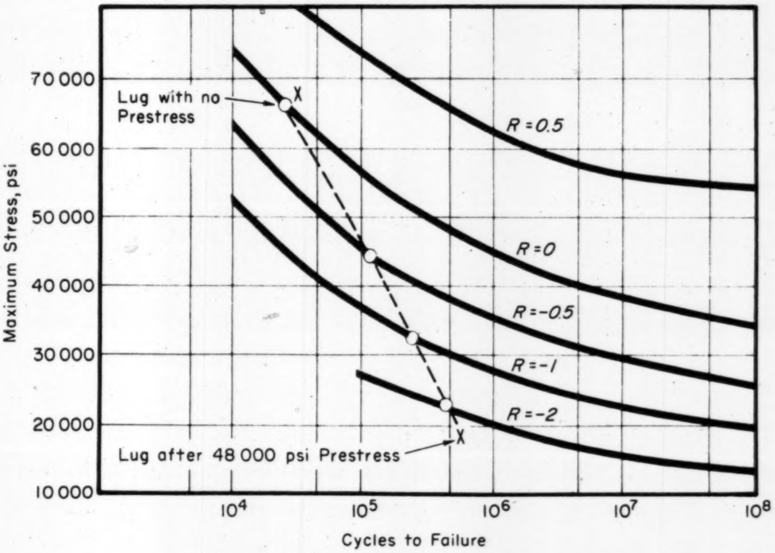


FIG. 9.—Effect of Residual Stress on Life.

Since only a very small portion of the cross-section suffers plastic deformation, this material will be forced into compression on unloading. The amount of compression will depend largely on the amount of plastic deformation. However, for small amounts, it can be assumed to be equal to the permanent set (strain between loading and unloading lines) times the modulus of elasticity. Where large amounts of plastic strain are involved, residual stresses equal to or greater than the yield stress of the material obviously could not be held. An arbitrary value of two-thirds the yield stress has proved to be a reasonable assumption.

---

[8] It was previously shown that the bushing was effectively twice as stiff as the high-stressed region of the lug. Accordingly, the combined spring constants would be the sum of both or three times that of the original lug. This is true whether or not both springs (lug and bushing) act in the same direction.

For estimating fatigue life after a load causing plastic deformation at the point of stress concentration, it is an easy matter to translate the whole stress program downward by the amount of residual stress. For example, if this residual stress amounted to 33,000 psi in compression, the lug, which formerly experienced 0 to 66,000 psi stress cycling, would now be subjected to ± 33,000 psi. Note that the stress range is the same in both cases, the manipulation being only a translation downward of the mean stress. Similarly, were the residual stress $-22,000$ psi, the corresponding cyclic stress would be from $-22,000$ psi to $+44,000$ psi for a stress range of 66,000 psi. Also, if the residual stress were $-44,000$ psi, the maximum stress would be $+22,000$ psi, and the new stress ratio would be -2 ($R = -2$).

We can now use the S-N curves shown in Fig. 6 to determine the fatigue life of our original lug for the various amounts of residual stress assumed in the last paragraph. These points are plotted in Fig. 9. Note the orderly fashion in which fatigue life seems to increase with lowering of mean stress (increase of residual stress). It should be remembered that these points represent the life of the same lug for the same loading condition, the only difference being the previous stress history; that is, it was assumed that the lugs were overloaded sufficiently to acquire residual compressive stresses in the above amounts.

We have shown that an increase in fatigue life can be expected in lugs having pressed-in bushings; also that an increase can be expected with static overloading prior to testing. However, overloading a lug having a pressed-in bushing would tend to loosen the bushing, which would shorten fatigue life, and at the same time it would produce beneficial residual compressive stresses, which would lengthen life.

Whether a net gain or loss could be expected would depend largely on the configuration, amount of bushing interference, and size of preload. In no case would the life be expected to be less than that predicted by a plot such as is shown in Fig. 9. Note that this particular plot is for a stress range of 66,000 psi; however, similar plots could be drawn for any stress range.

One set of lugs having a 0.0075-in. interference was prestressed to 48,000 psi prior to cycling at 18,800 psi nominal stress. The average life was 541,000 cycles (average of four specimens). Assuming that this overload was sufficient to relieve all of the interference, it appears as though the stress ratio was in the neighborhood of -2.2, and the residual stress would have to have been about $-44,500$ psi.[9] This point is plotted in Fig. 9.

Unfortunately, no other experiments were made on static overloading of lugs with pressed-in bushings. The linear strain theory would predict that a 38,000-psi prestress would have been sufficient to relieve all of the bushing interference, whereupon the subsequent life would have been 400,000 cycles. This would represent the greatest loss to be expected for this configuration. Greater amounts of overloading would increase life by producing additional beneficial residual compressive stresses, and lesser amounts would not completely relieve the benefits of pressed-in bushings. It should be noted that 400,000 cycles is substantially greater than the life for a lug without a bushing.

---

[9] According to the assumptions made in the linear strain theory, a 48,000 psi preload should have induced a residual stress higher than the yield strength of the material. Therefore, a further assumption was made that only $\frac{2}{3}$ of this amount could be sustained, and a calculated residual stress would have been $\frac{2}{3}(-70,000) = -46,000$ psi instead of the $-44,500$ psi shown above. These differences, however, would not have materially changed the predicted fatigue life.

So far, effects of unequal distribution of loads, interference fits, and effects of prestressing have been discussed. In an effort to combine the benefits of the interference fit and at the same time to make the first fastener in a joint incapable of carrying too much load, splices were tested with end rivets replaced by $\frac{1}{4}$-in. diameter tapered bolts having 0.003-in. interference on the skin side and 0.032 in. clearance on the doubler. These data are compared with those for riveted joints in Fig. 10. Note that there seems to be a lesser improvement at the lower stress levels than at the upper, indicating that a lesser interference could have been used for lower-level loading.

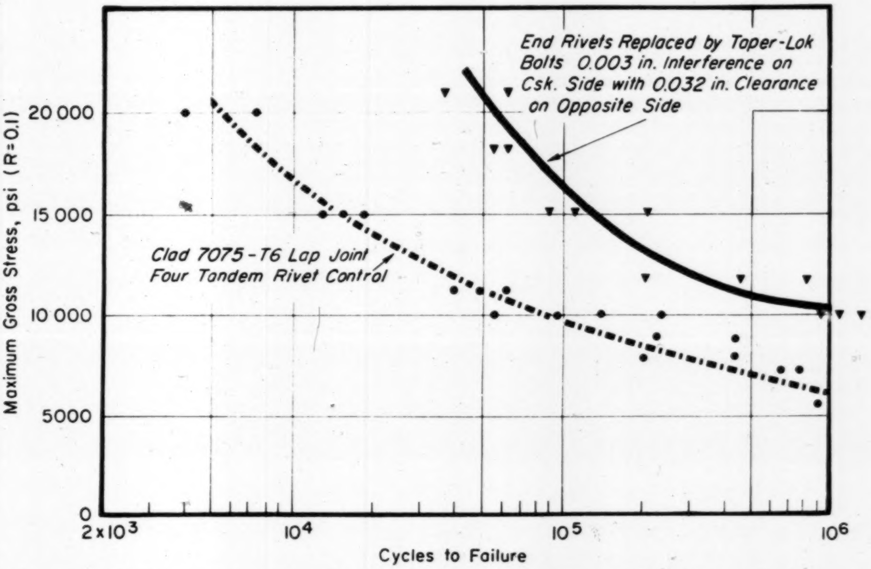


FIG. 10.—*S-N* Curves for Four Rivet Lap Joints and Joints with Interference-Fit Bolts.

SUMMARY

In comparing the effective spring constant of a pressed-in bushing with that of the surrounding structure, the assumption was made that the nominal rate of contraction of the bushing could be used for comparing localized strains at the point of concentration. The fact that good agreement was obtained with test data seems to indicate that these assumptions were nearly correct, although it appears that this could occur only if the bushing and surrounding structure acted as a unit at these points. An alternate explanation might be that the nominal strains in the bushing at points away from the concentration neither contributed to nor subtracted from the local strains.

While the method described in this paper may lend itself to use with riveted structures, considerable testing would be required for obtaining necessary spring-constant ratios and effects of overloading. Initial losses and subsequent gains in fatigue life could probably be represented by families of curves superimposed on those already shown in Figs. 7 and 9. However, this falls beyond the scope of the present paper, and could be accomplished only with much more extensive research.

CONCLUSION

A method for determining the effect of interference fits on fatigue strength has been presented. While not altogether rigorous, good agreement with test data was obtained. More work needs to be done regarding loss of fatigue strength with overloads, especially towards obtaining structures incapable of inducing loads causing these losses.

A mechanism such as described, or one very similar, should be useful for estimating cumulative damage in fatigue. However, more work would be required for necessary spring-constant ratios of rivets or other fasteners to their surrounding structures.

Acknowledgments:

The author wishes to express his appreciation to Mr. G. D. Lindeneau and other members of the Convair Structures Laboratory for valuable assistance in obtaining data. He also wishes to thank Mr. D. M. Forney, Jr., and the Materials Laboratory of Wright Aeronautical Development Div. for permission to use photographs of plastic models from Contract No. AF33(616)-6293. Plastics for models were furnished by Swedlow Inc., Los Angeles, Calif. Tapered bolts (Taper-Lok) were furnished by the Briles Mfg. Co., El Segundo, Calif.

REFERENCES

(1) C. R. Smith and G. D. Lindeneau, "Riveted Joints—Fatigue Strength," Papers on Fatigue of Aircraft Structures, *ASTM STP No. 203*, Am. Soc. Testing Mats., p. 10 (1956).

(2) W. D. Mitchell and D. Rosenthal, "Influence of Elastic Constants on the Partition of Load Between Rivets," *Proceedings*, Soc. Experimental Stress Analysis, Vol. VII, No. 2, p. 17 (1949).

(3) C. R. Smith, "Thin Doublers for Fatigue-Resistant Aluminum-Alloy Structures," *Aero-Space Engineering*, Feb., 1959, p. 40.

(4) F. M. Howell and J. L. Miller, "Axial-Stress Fatigue Strengths of Several Structural Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 955 (1955).

(5) H. T. Jessop, C. Snell and G. S. Holister, "Photoelastic Investigation on Plates with Single Interference-Fit Pins with Load Applied (*a*) to Pin Only, and (*b*) to Pin and Plate Simultaneously," *The Aeronautical Quarterly*, Vol. IX, May, 1958, p. 155.

(6) C. R. Smith, "Prediction of Fatigue Failures in Aluminum Alloy Structures," *Proceedings*, Soc. Experimental Stress Analysis, Vol. XII, No. 2, p. 21 (1955).

DISCUSSION

Mr. Eric Aubrey.[1]—Has the author conducted any tests with a clearance hole instead of an interference fit? Some investigations have shown that below a certain interference fit, its effect on fatigue strength is small.

Mr. C. R. Smith (*author*).—For the first part of your question, the answer is no. For the second part, I believe the references you mention were based on tests at high mean stresses, something on the order of 1 ± 0.4 g, to simulate airplane gust loading. Such a test on the lug shown in Fig. 5 would show no increase in life for the 0.005-in. interference because the effect of interference is already had in the loading. This points to the importance of including ground-to-air cycles for evaluating the fatigue life of airplane structures.

Mr. Edwin J. Zapel.[2]—It is rather interesting to note that in this paper the author has shown that the influence of residual stress, whether induced by plastic deformation or by interference from a lug, effectively distributes the loading of the lug or of the load-carrying part in such a manner that the material in the most critical region is no longer the most critical material. That is exactly what has been done. In the case of the joints shown in the plastic models the same effect could be obtained perhaps in some other manner, although that is by far the best way to do it.

We have done some work in our photoelastic laboratory in much the same way, and I am quite pleased to find some correlation with our own information.

Mr. Smith.—I do not know that I have anything to add to that. Perhaps the statement on residual stress should be clarified in that the residual stresses described here were in tension and, as such, are never beneficial. The benefit lies in the fact that they enable the structure to take advantage of the added stiffness of bushing or bolt. This lowers the stress amplitude, not necessarily the peak stress.

---

[1] Group Leader, Structures Development, Canadair Ltd., Montreal, Quebec, Canada.

[2] Research Specialist, Boeing Airplane Co., Seattle, Wash.

THE FATIGUE NOTCH SENSITIVITY OF ANNEALED COPPER*

By J. Y. Mann[1]

Synopsis

Rotating-cantilever fatigue tests are reported on notched annealed copper specimens having K_t values of 1.5, 2.5, 4.0, 5.5, and > 10.

Contrary to the commonly accepted view, this investigation has shown that annealed copper is not particularly fatigue-notch-sensitive—the maximum K_f value found being less than 1.5.

There is also evidence that this material is unlikely to exhibit the phenomenon of "nonpropagating cracks."

There is abundant evidence, both from laboratory tests and service experience, that stress concentrators can produce premature failure in components subjected to fatigue loadings. The magnitude of the fatigue strength reduction under a notch of particular theoretical stress concentration factor, K_t, depends, among other things, upon the material considered, the loading conditions, and the stress range to which the specimen is subjected. In most cases the numerical value of the fatigue strength reduction factor, K_f, is less than the theoretical factor, K_t, although wrought metallic materials under rotating-bending conditions apparently exhibit K_f values close to the chosen value of K_t until the latter is within the range 2 to 4. It is only at K_t values exceeding this range that the full theoretical factor is not realized. There is also evidence that the fatigue strength reduction caused by notches of K_t exceeding about 5 may be less than those of lower K_t (**1**)[2] and that such notches exhibit the phenomenon of "nonpropagating cracks" (**2**).

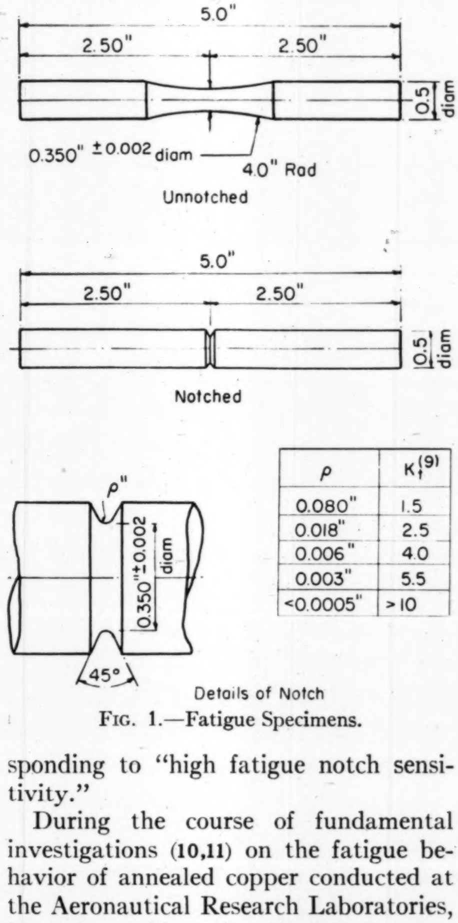
One explanation for the differences in susceptibility of various materials to notches of the same K_t is based on the assumption that in the more ductile materials plastic deformation occurs at the base of the notch and that this tends to reduce the peak stress. This would suggest that a material which exhibits a high degree of ductility, as indicated for example by a tension test, may be relatively notch-insensitive in fatigue. Copper was one of the first non-ferrous metals on which fatigue tests were reported (**3**), and in the annealed condition it is highly ductile, yet it has been stated in several papers (**4**–**7**) that annealed copper is markedly notch-sensitive in fatigue. The origin of this belief is not clear, although it seems probable that it is based on the results of an investigation on a number of materials reported by Moore and Lyon in 1927 (**8**). These investigators reported that for a notch of Neuber (**9**) $K_t = 1.75$ annealed copper indicated a fatigue strength reduction factor of about 1.7—equal to the greatest fatigue strength reduction of any of the materials which they examined. However, the notch used by Moore and Lyon cannot be regarded as particularly severe and, compared with some materials which exhibit K_f values of 5 or greater under certain severities of notch, a value of $K_f = 1.7$ cannot be regarded as corresponding to "high fatigue notch sensitivity."

---

* This paper is published with the permission of the Chief Scientist, Australian Defence Scientific Service, Department of Supply, Melbourne, Australia.

[1] Scientific Officer, Australian Defence Scientific Service, Aeronautical Research Laboratories, Department of Supply, Melbourne, Australia.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.



ρ	K_t (9)
0.080"	1.5
0.018"	2.5
0.006"	4.0
0.003"	5.5
<0.0005"	>10

FIG. 1.—Fatigue Specimens.

During the course of fundamental investigations (**10,11**) on the fatigue behavior of annealed copper conducted at the Aeronautical Research Laboratories, it became evident that the fatigue properties of this material were insensitive to methods of surface preparation and structural inhomogeneities. In view of the supposed "high fatigue notch sensitivity" of annealed copper it was decided to investigate the notched fatigue properties of this material more fully and to carry out a series of tests complementary to those on 24S-T aluminum alloy which have already been fully reported (**12**).

MATERIAL AND SPECIMENS

The material used in this investigation was refined high-conductivity electrolytic copper (of purity not less than 99.85 per cent) and was received in the form of $\frac{5}{8}$-in. diameter extruded bar. Two specimens from each bar were made into tension specimens while the remainder of each bar was sufficient for the manufacture of at least 30 fatigue specimens.

TABLE I.—TENSILE PROPERTIES OF MATERIAL.

	As-received	Annealed
0.1 per cent proof stress, psi	34 500	2 500
0.2 per cent proof stress, psi	35 500	2 800
Ultimate tensile strength, psi	37 000	31 000
Reduction of area, per cent	86	87
Elongation in 2 in., per cent	30	61
Vickers diamond hardness, 2½ kg load	95	38

Unnotched and notched fatigue specimens, the general form of which are shown in Fig. 1, were fully machined from the extruded bars. After cutting to length, unnotched blanks were rough-machined to approximately 0.030 in. oversize on all diameters. These were then turned to size taking three 0.005-in. and one 0.001-in. deep cuts at feeds of 0.007 in. and 0.004 in. per revolution respectively. The final finishing cuts were made using a carbide tipped tool having a tip radius of about 0.025 in. Notched blanks were turned to size on the cylindrical surface and the notches then plunge-cut using circular form tools of appropriate tip radii to give K_t (**9**) values of 1.5, 2.5, 4.0, 5.5, and >10. Lathe

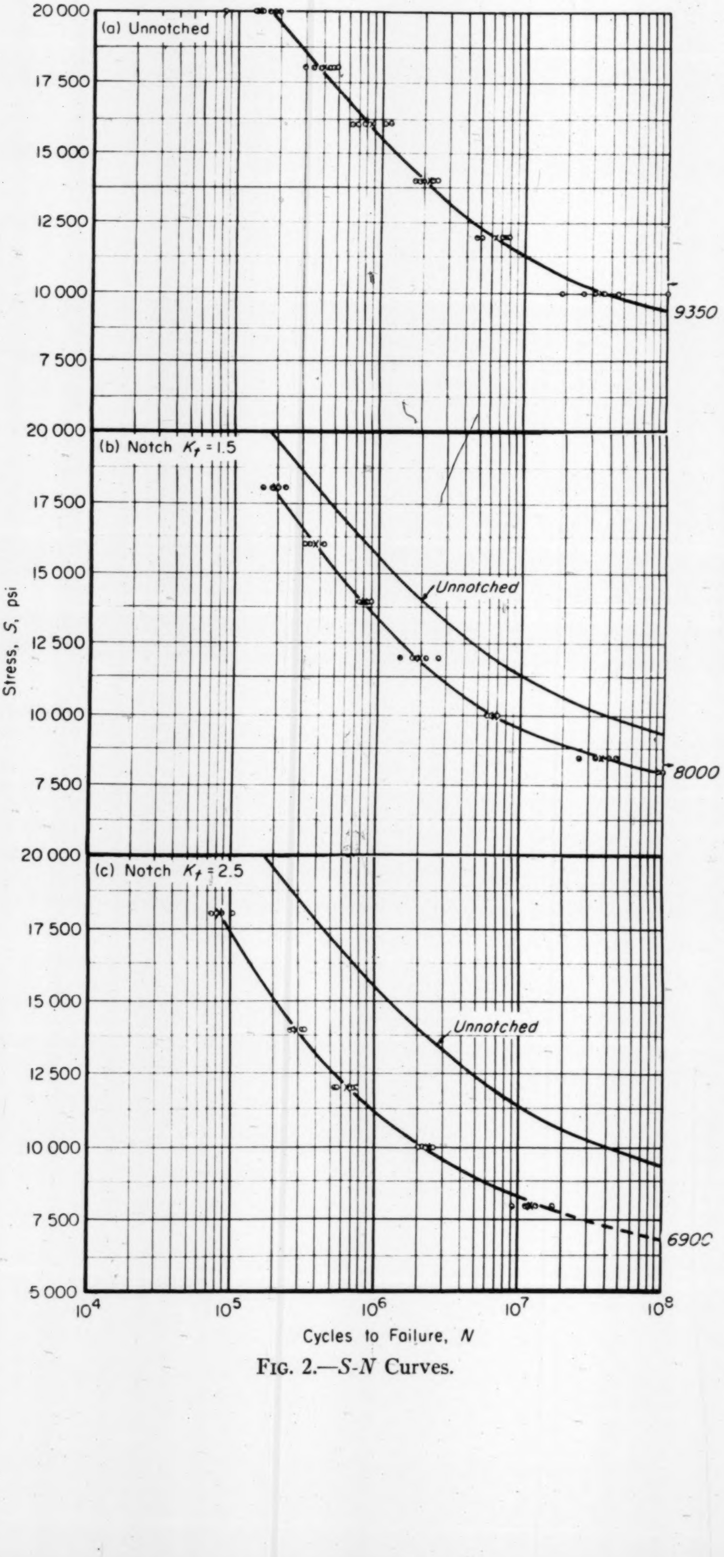


FIG. 2.—*S-N* Curves.

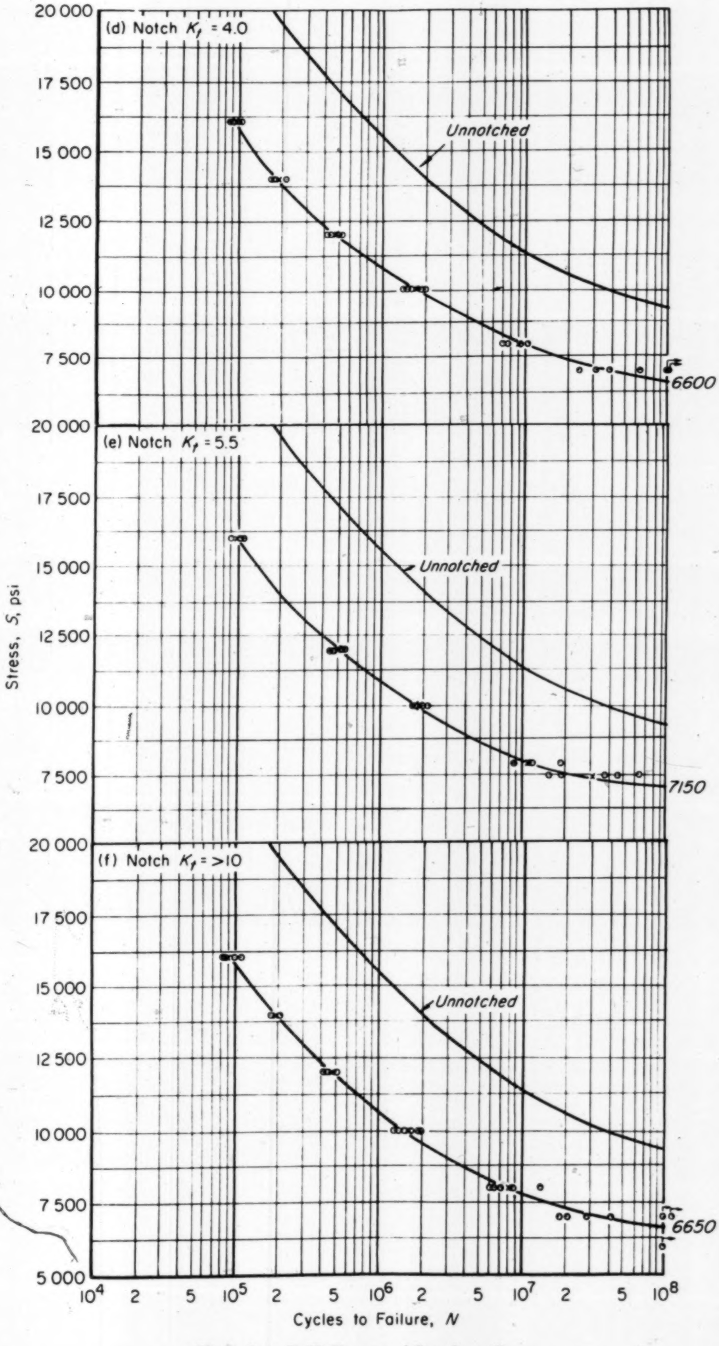


FIG. 2.—*S-N* Curves (*Continued*).

spindle speed during the finishing operations was 450 rpm, a copious supply of soluble oil being used as a coolant and lubricant during all machining operations. After machining, the specimens were degreased and vacuum annealed (approximately 10^{-3} mm of mercury) in batches of seven for 45 min at 650 C, followed by slow cooling to room temperature in the evacuated enclosure.

TABLE II.—MAXIMUM STRENGTH REDUCTION FACTORS.

K_t	Maximum K_f (K_{fm}) at Cycles Indicated		$\frac{K_{fm}}{K_t}$, per cent
1	1	...	100
1.5	1.20	8×10^6	80
2.5	1.39	2×10^6	55
4.0	1.44	7×10^5	35
5.5	1.43	7×10^5	25
>10	1.46	2×10^6	15

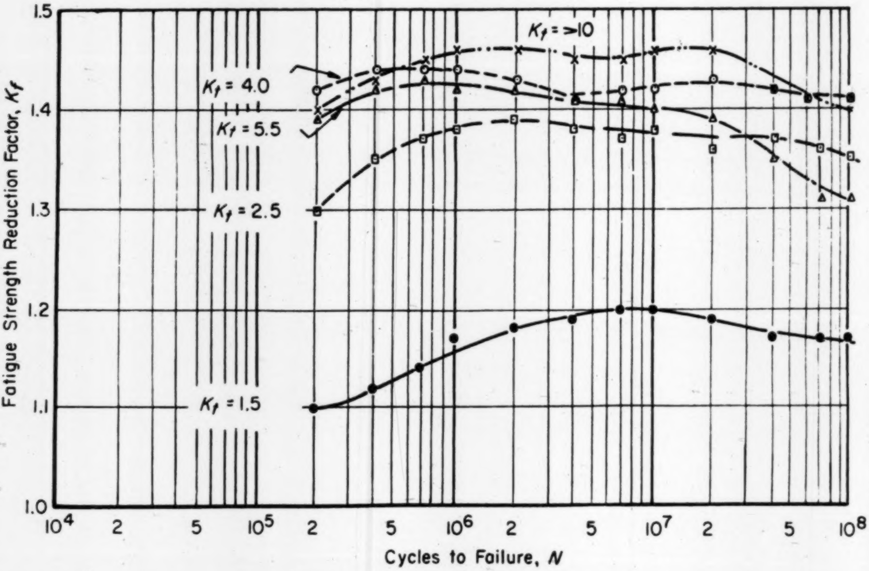


FIG. 3.—Variation of K_f with Endurance.

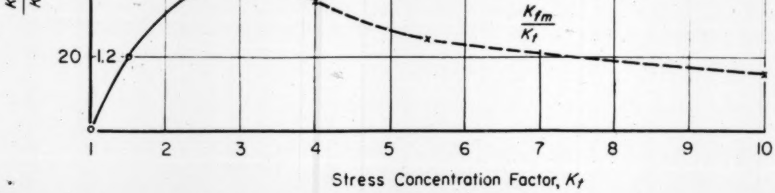


FIG. 4.—Relation Between K_{fm} and K_t.

Under these conditions no visible scaling occurred and the grain size developed was about 0.1 mm (0.004 in.). The test sections of finished specimens were measured using a toolmakers' microscope.

Tension tests were made on "as-received" and annealed specimens from each bar, and the average results of these, together with hardness tests, are given in Table I.

FATIGUE TESTS

All fatigue tests were carried out in two of the Aeronautical Research Laboratories single-point-loading rotating-cantilever fatigue machines which, for this investigation, were operated at a frequency of 6000 cpm instead of that normally used, that is, 12,000 cpm. This was found necessary in order to prevent permanent deformation of the specimen while running through a critical frequency. Approximately 200 specimens were used for this investigation, between 30 and 36 specimens being tested to determine each *S-N* curve, that is, generally six specimens at each chosen stress level. Failure was defined as complete fracture of the specimen.

Figure 2(*a* to *f*) show the results of fatigue tests together with the logarithmic mean *S-N* curves for each notch. The strength reduction factors, K_f, for each notch at different endurances were calculated from these mean *S-N* curves and curves of K_f against log N, so obtained, are shown in Fig. 3. Table II and Fig. 4 indicate the maximum fatigue strength reduction factor, K_{fm}, resulting from each notch and its percentage of the theoretical stress concentration factor.

A surprising feature in these fatigue tests is the small "scatter" in endurances, particularly in the case of notched specimens. In one instance (notch $K_t = 1.5$, 10,000 psi) the six specimens tested failed between endurances of 6×10^6 and 7×10^6 cycles.

DISCUSSION

In the annealed condition, the copper used in this investigation was extremely soft and ductile, as Table I indicates, and all fatigue specimens were tested at nominal stresses exceeding the 0.1 per cent proof stress values. It is apparent from this investigation that, contrary to the commonly accepted view (**4–8**), annealed copper is not particularly fatigue notch sensitive as the maximum strength reduction factor indicated is less than 1.5. In fact, the maximum strength reduction factors for notches of $K_t = 2.5$, 4.0, 5.5, and >10 range between 1.4 and 1.45. This result supports the view (**13,14**) that low yield stress - ultimate tensile stress (or proof stress - ultimate tensile stress) ratios are associated with reduced fatigue notch sensitivity, and that "ductility" is an important criterion in improved fatigue resistance.

Metallographic examination of longitudinally-sectioned notched specimens which had endured 100×10^6 cycles without fracture revealed cracks in the notches. However, as 15 specimens failed at endurances between 20×10^6 and 95×10^6 cycles, it seems unlikely that these were "nonpropagating cracks." The close agreement between the *S-N* curves for notches of $K_t = 4.0$, 5.5, and >10 also suggests that annealed copper may not exhibit the phenomena of a critical stress concentration factor and "nonpropagating cracks." This conclusion is contrary to that of Frost and Dugdale (**15**) who carried out axial loading fatigue tests on sheet specimens of annealed copper and reported finding a "nonpropagating crack" in a specimen which had endured about 27×10^6 cycles. On the basis of evidence discussed above, and investigations on fatigue crack propagation reported by these authors (**15**) and others (**16,17**) which suggest that crack propagation is an erratic process involving periods of no apparent growth, there seems little justification in the assumption that the crack formed in one of their copper specimens was "nonpropagating."

In previous papers by the author on the fatigue properties of 24S-T aluminum alloy (**1**,**12**), it was suggested that a simple linear relationship might exist between $\frac{K_{fm}}{K_t}$ and K_t. The results of this investigation on annealed copper do not support this proposal, although it may prove to be a useful approximation in the case of the more usual structural and constructional materials.

CONCLUSIONS

1. Contrary to the commonly accepted view, annealed copper is not particularly fatigue notch sensitive—the maximum K_f value found being less than 1.5.

2. It appears unlikely that annealed copper will exhibit the phenomenon of "nonpropagating cracks."

Acknowledgments:

The author's thanks are extended to Mr. M. M. Hutchison, who carried out the vacuum annealing of all specimens used in this investigation, and to Mr. A. G. Cole, who was responsible for the conduct of the tension tests.

REFERENCES

(**1**) J. Y. Mann, "The Effect of Stress Concentrations on the Fatigue Resistance of a Duralumin Type Aluminum Alloy," *Journal*, Royal Aeronautical Soc., Vol. 60, No. 550, pp. 681–685, Oct., 1956.

(**2**) N. E. Frost, "Non-Propagating Cracks in Notched Specimens Subject to Fatigue Loading," *Aeronautical Quarterly*, Vol. 8, Part 1, pp. 1–20, Feb., 1957.

(**3**) A. Wöhler, "Resultate in der Central-Werkstatt der Nieder-schlesisch-Märkischen Eisenbahn zu Frankfurt a.d.O. angestellten Versuche über die relative Festigkeit von Eisen, Stahl und Kupfer," *Zeitschrift für Bauwesen*, Vol. 16, pp. 67–83 (1866).

(**4**) Battelle Memorial Institute, "Prevention of the Failure of Metals Under Repeated Stress," John Wiley & Sons, Inc., New York, N. Y., p. 212 (1941).

(**5**) R. G. Wilkinson and J. B. Wilson, discussion of paper by H. J. Pollard, "New Materials and Methods for Aircraft Construction," *Journal*, Royal Aeronautical Soc., Vol. 57, No. 509, May, 1953, p. 297.

(**6**) G. A. Cottell, "Fatigue Failures with Special Reference to Fracture Characteristics," *Technical Report* (New Series), British Engine Boiler & Electrical Insurance Co., Ltd., Vol. 2, Sept., 1954, p. 246.

(**7**) P. Forrest, "The Effect of Temperature Upon the Fatigue Properties of Steel," *Metal Fatigue*, J. A. Pope, editor, Chapman and Hall, Ltd., London, p. 172 (1959).

(**8**) H. F. Moore and S. W. Lyon, "Tests of the Endurance of Gray Cast Iron under Repeated Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part 2, pp. 93–94 (1927).

(**9**) H. Neuber, "Theory of Notch Stresses," J. W. Edwards, Ann Arbor, Mich. (1946).

(**10**) R. B. Davies, J. Y. Mann and D. S. Kemsley, "Hardness Changes During Fatigue Tests on Copper," *Proceedings*, International Conference on Fatigue of Metals, London, Institution Mechanical Eng., pp. 551–556 (1956).

(**11**) J. Y. Mann and R. B. Davies, "Some Variations in the Fatigue Behavior of Low-Oxygen Copper Bar," Report, Dept. Supply, Aeronautical Research Laboratories (To be published).

(**12**) J. Y. Mann, "The Effect of Stress Concentrations on the Fatigue Resistance of 24S-T Aluminium Alloy," Report A.R.L./SM. 217, Dept. Supply, Aeronautical Research Laboratories, Nov., 1953.

(**13**) C. A. Stubbington and P. J. E. Forsyth, "Some Observations on the Air and Corrosion Fatigue Behavior of a Ternary Aluminium-Zinc-Magnesium Alloy" (Interim Report), R. A. E. Technical Note Met. 258, March 1957.

(**14**) W. A. P. Fisher, "Experimental Correlation Between the Endurance of a Wing Spar

Joint and the Ratio Between 0.1 per cent Proof and Ultimate Tensile Strengths of the Material," Current Paper 371, Aeronautical Research Council (1958).

(15) N. E. Frost and D. S. Dugdale, "The Propagation of Fatigue Cracks in Sheet Specimens," *Journal of Mechanics and Physics of Solids*, Vol. 6, No. 2, pp. 92–110 (1958).

(16) M. S. Hunter and W. G. Fricke, "Cracking of Notch Fatigue Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 643 (1957).

(17) H. A. Lipsitt, F. W. Forbes and R. B. Baird, "Crack Propagation in Cold-Rolled Aluminum Sheet," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, pp. 734–754 (1959).

STATIC AND FATIGUE STRENGTH OF METALS SUBJECTED TO TRIAXIAL STRESSES*

By William M. Lehrer[1] and Harry Schwartzbart[1]

Synopsis

The objectives of this program were: (1) the determination of the effect of triaxial stresses on fracture in static and dynamic loading and (2) determination of the brittle fracture strength of metal in the absence of any preceding plastic flow. To achieve these objectives, use was made of a composite brazed specimen consisting of a thin disk of soft metal between cylinders of hard metal. When a load is applied to the brazed bars, a triaxial stress condition is created due to the plastic restraint on the metal by the harder steel cylinder interfaces.

Experimental procedures and equipment were developed for producing sound and voidless brazements over a range of joint thicknesses, for accurately measuring joint thickness and for determining the tensile and fatigue properties of such brazements with a minimum of scatter.

The static tensile strength of silver butt brazements in 4340 steel increases with decreasing joint thickness because of increasing triaxiality of stress, reaches a maximum at a joint thickness of 1.5×10^{-4} in., and falls to a relatively constant value of 26,000 psi at thicknesses less than 4×10^{-5} in. The maximum strength is greater than 110,000 psi, some 7½ times the strength of cast silver in uniaxial tension. The ultimate tensile strength of pure cast silver has been determined to be approximately 15,000 psi.

The fatigue life of silver-brazed butt joints in 4340 steel increases continuously with decreasing joint thickness for both fluctuating tension and completely reversed axial loading over the entire range of joint thicknesses investigated. 3×10^{-4} in. to 1.8×10^{-2} in. Also determined and included with these data are the fatigue properties of pure cast silver to simulate the infinitely thick joint.

Relationships between the apppearance of the fracture surfaces and the mechanical properties are drawn, where possible.

One of the most promising possibilities of inducing triaxial tensile stresses in a given material was first discussed by Orowan in 1946 (**1**).[2] The test described involves a thin cylindrical disk bonded between two cylindrical pieces of much higher strength. The composite cylindrical specimen is pulled in tension. The sandwiched disk reaches its normal flow stress sooner than the remainder of the specimen but is prevented from yielding by virtue of its being bonded to the harder cylinders. This gives rise to transverse tensions, and a state of triaxial tension is obtained. In the limiting case of an infinitely thin disk between cylinders of higher yield strength than the fracture strength of the disk, very high states of triaxiality may be realized.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Armour Research Foundation of Illinois Institute of Technology, Chicago, Ill; Mr. Lehrer is now with Raytheon Manufacturing Co.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Bredzs (2), in a study of the factors influencing the strength of brazed joints, illustrated a brazing technique for the preparation of such specimens. It was shown in this study that pure silver, when bonded between cylinders of low-carbon steel, failed through the plane of symmetry of the silver disk at loads greatly in excess of the uniaxial tensile strength of the silver, provided the joint was sufficiently thin. This work was further elaborated upon in a later paper by Bredzs and Schwartzbart (3) in which the mechanical aspects of this composite brazed specimen were discussed. This paper indicated that the three sources of the circumferential and radial stress developed in the sandwiched metal are: (*a*) different coefficients of expansion of filler and parent metal, giving rise to stresses on cooling from the brazing operation; (*b*) different moduli of elasticity of filler and parent metal, giving rise to stresses entirely within the elastic ranges of either metal; and (*c*) different yield stresses of filler and parent metal. It was pointed out that the last is the principal source of transverse stresses.

The strength of the silver joint between harder cylinders has also been investigated by Moffatt and Wulff (4). These investigators have shown experimentally that the development of triaxial tensile stresses is not only a function of joint thickness but is also a linear function of the joint thickness to diameter ratio. Yoshida (5) has arrived at a quantitative formula showing the relationship between the strength and both the radius and the thickness of the joint. This quantitative formula presents, however, the fracture strength of the joint as a function not of $t/2R$, according to Moffatt and Wulff, but as a function of t^2/R^2 where t and R are the thickness and radius of the disk, respectively. Through his analysis, Yoshida further concludes that the fracture strength of the disk can, as a limiting case, exceed the fracture strength of the base material.

Bühler and Colbus (6) have performed experiments with ingot iron, tin, and lead as the filler metal and have found that the relationship of t/D^2 to the log of the fracture stress is a straight line only over certain increments of the entire curve. As the ratio decreases, inexplicable breaks in the curve to greater strength occur. The breaks in the curve occur for the three filler metals used—the greater the bulk strength, the lower the t/D ratio before the slope changes.

It was the object of this study to obtain quantitative data relating the disk thickness and the fracture strength under both static tensile loading and dynamic loading. It was desired to study mechanical effects only, as independently as possible of complicating metallurgical reactions between base metal and filler metal. This required judicious selection of filler metal and base metal, development of techniques for the brazing of the selected base material with the given filler metal through a wide range of joint thicknesses, development or adaptation of a means of accurately measuring the thickness of the resulting brazed joints, and the construction of axial loading fixtures for the tensile and fatigue testing of the brazements.

SELECTION OF FILLER METAL AND BASE METAL

Basic Factors:

The choices of filler and base metals to be studied were contingent on several considerations:

1. It was desired that there be no metallurgical interaction between base and filler metals so that only mechanical effects could be observed.

2. The yield strength of the base metal

was to be higher than the fracture strength of the filler metal so that no plastic deformation would occur in the base metal.

3. Of prime importance was the ability of the filler metal to wet and braze the base metal, preferably without fluxes which might weaken the joint through entrapments.

4. Because of the relationship between crystalline lattice structure and the propensity of a metal to exhibit a ductile-to-brittle transition, it was desirable to include filler materials of lattice structures other than body-centered cubic.

These considerations led to the choices of steel as base metal, and as filler metals silver and lead, both face-centered cubic. Virtually no mutual solubility exists between these filler metals and iron. Mild steel was chosen as the base metal with lead filler, while 4340 steel was utilized with silver filler metal.

Wettability of Lead on SAE 1018 Steel:

Bredzs (2) has shown that the surface oxides of 1018 steel may be reduced quite satisfactorily with inexpensive, nonexplosive commercial tank 10 per cent H_2 + 90 per cent N_2. On this basis, this gas was selected for the wettability tests of lead on low-carbon steel.

According to Bailey and Watkins (7), the contact angle between molten lead and solid iron is greater than 90 deg, indicating a nonwetting condition. This was borne out in sessile drop experiments in which no spreading of the lead occurred even at temperatures up to 1800 F with complete reduction of the steel surface oxides.

However, according to the above work, the addition to the lead of a small amount of nickel should increase wettability on iron. Accordingly, lead alloys were prepared containing 0.1 per cent nickel and 0.2 per cent nickel and their wettability determined with a sessile drop on low-carbon steel bar. It was found that the addition of 0.2 per cent nickel to pure lead would decrease the contact angle sufficiently to produce sound, thin, lead-brazed joints in low-carbon steel.

Wettability of Silver on 4340 Steel:

Preliminary studies of the strength of extremely thin silver joints in drill rod as reported by Bredzs (2) reveal strengths approaching 85,000 psi. Since one condition leading to the development of maximum triaxiality is a base metal yield strength exceeding the fracture strength of the filler metal, low-carbon steel bars could not be used as the base metal for the silver-brazed joints.

To achieve the higher required yield strength, SAE 4340 steel was selected as the base material to be used with silver. The fact that SAE 4340 steel contains chromium necessitated the substitution of pure dried hydrogen for tank 90 per cent N_2 + 10 per cent H_2 to reduce the refractory chromium oxides.

The hydrogen used was of commercial tank purity containing moisture to a dew point of approximately −30 F and small traces of oxygen. The purification and drying train consisted of two palladium-platinum catalyst purifiers. The bulk of the moisture formed during the catalytic reduction of the O_2 present was absorbed in an indicating $CaCl_2$ bed and the remaining moisture then collected in a liquid nitrogen cold trap.

In sessile drop tests using the above reducing atmosphere in the bell jar, the silver was noted to flow and wet to a degree sufficient to fill a minimum capillary gap in 4340 steel.

EXPERIMENTAL PROCEDURES AND EQUIPMENT

The steel bars used for the preparation of the butt-brazed tension specimens and fatigue specimens measured 3 in. long × $\frac{3}{4}$ in. diameter to result in a 6 in. long brazed specimen. The faying surfaces of the bars were surface-ground plane and perpendicular to the bar axis in a specially designed fixture. Following surface grinding, the surfaces to be mated were automatically polished to optical flatness with No. 1900 Al_2O_3 grit. Details of the grinding and polishing procedures and equipment will be found in reference (**8**). After polishing, a groove was filed in the end of the bar so as to form a wedge-shaped cup whose apex, when in contact with the mating surface, was at the capillary to be filled with the molten filler metal. Prior to brazing, the bar ends to be brazed were wiped clean with carbon tetrachloride.

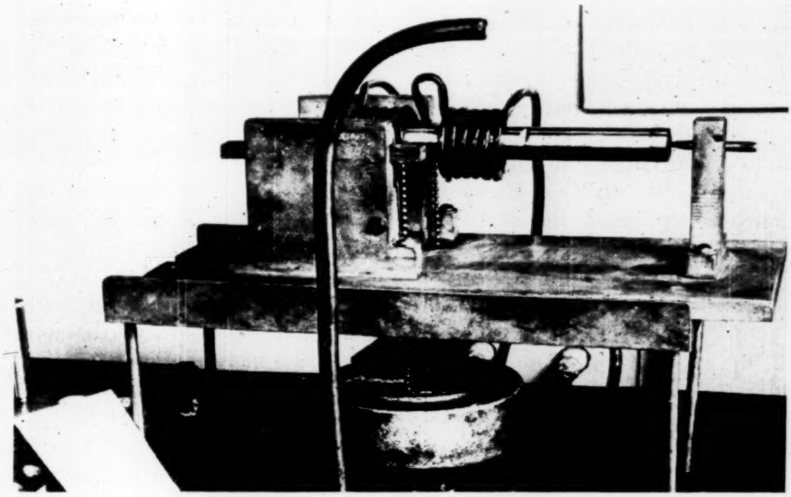


Fig. 1.—Brazing Apparatus in Loading Position.

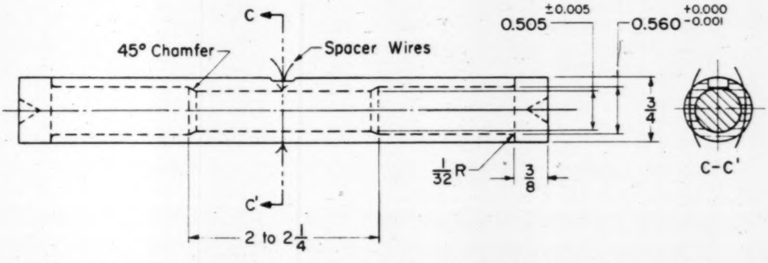


Fig. 2.—Proper Arrangement of Spacer Wires Between Half-Bars and Dimensions of Tension Specimen After Machining the Brazed Bar (Tension Specimen Outlined with Dashed Lines).

The brazing apparatus was an adaptation of that used by Bredzs (**2**) and is shown in Fig. 1. Not shown in the figure is the covering bell jar allowing brazing in pure dry H_2 or H_2-N_2 mixture or the nugget of filler alloy sitting in the cup formed by the two filed edges. After loading the brazing apparatus with the half-bars, the nugget of metal, and suitable weight to insure contact of the faying surfaces, the apparatus was slid into the work coil of a 15-kw spark gap generator. After purging the system of oxygen, the braze was made with induction heating.

By shimming with molybdenum spacer wires, various joint thicknesses were achieved. Molybdenum was chosen as the spacer material due to its insolubility in molten silver and molten lead and its resistance to deformation at brazing temperatures. In order not to interfere with the capillary forces of the filler metal, the spacers did not touch the feeding cup. The best procedure was to place them symmetrically on the left and right of the feeding cup as is illustrated in section *C-C′* of Fig. 2. For obtaining very thin joints, no spacer wires were used and the bars merely butted directly against each other. The half-bars used for the shimless brazes had a feeding cup on both top and bottom in an effort to distribute the longitudinal supporting forces on the joint surfaces more uniformly. This procedure resulted in a more uniform thickness of the thin filler disk.

The tension specimen prepared from the brazed test bars is shown by the dotted lines in Fig. 2. The button-head specimen was ground at the shoulders to better than a No. 16 finish and held to a tolerance of ±0.0005 in. The fatigue bars fabricated from the brazed test bars were simple cylinders of both $\frac{1}{4}$ and $\frac{1}{2}$-in. diameter and 6 in. long. These bars were fabricated by centerless grinding to a No. 32 finish and, again, a maximum of $\frac{1}{2}$ mil tolerance.

Two methods were utilized in this study to determine accurately the thickness of the brazed joint. These were use of the optical microscope for thick joints and electrolytic stripping of the filler metal after fracture for joints thinner than could easily be resolved microscopically.

The optical method was that used by Bredzs (2). After fabricating the brazed bar to the tension or fatigue specimen, measurements were taken at 90-deg intervals around the periphery of the joint with a portable microscope fitted with a filar ocular calibrated in microns. Despite careful polishing of the surfaces and contrasting etchant, joints less than 0.003 in. thickness were found to be too thin for accurate optical measurement and, therefore, had to be measured by electrolytic stripping.

A practical electrolytic stripping device was that developed by Francis (9) and adapted to the needs of this study. It was possible to dissolve silver and lead anodically from the steel base metal after fracture in solutions of KCN and NaOH, respectively. After calibration of the solutions and instrument, application of Faraday's law resulted in an easy and accurate calculation of the joint thickness. The data required for the calculation were the equivalent weight of the coating metal, the area of the surface stripped, the current used in stripping, and the time required for stripping. These latter two data were provided by the Francis thickness tester. The calculation assumed 100 per cent coverage of the joint area and the density of the filler metal to be that of as-cast metal. The error introduced through these assumptions will be discussed later in the paper. Complete details of the calibration of the electrolytic thickness tester are included in reference (8). The accuracy of measurement of visually perfect joints was within 2 per cent and the sensitivity below 1 microinch of thickness.

In an effort to reduce bending or tearing stresses in the brazed joint and to maintain axial loading on the specimen, a concentric loading tension testing fixture was constructed after that used by G. Sachs et al (10) and by the National Advisory Committee for Aeronautics (11). Concentricity is attained in the fixture principally due to the tension specimen being loaded between two points which are the points of contact between the upper and lower ball and plane. Provided that the tolerances on all precision components are kept at a minimum (±0.0005), the fixture and assembled specimens have essentially inherent concentricity.

The fatigue fixture utilized in this work was that especially designed by

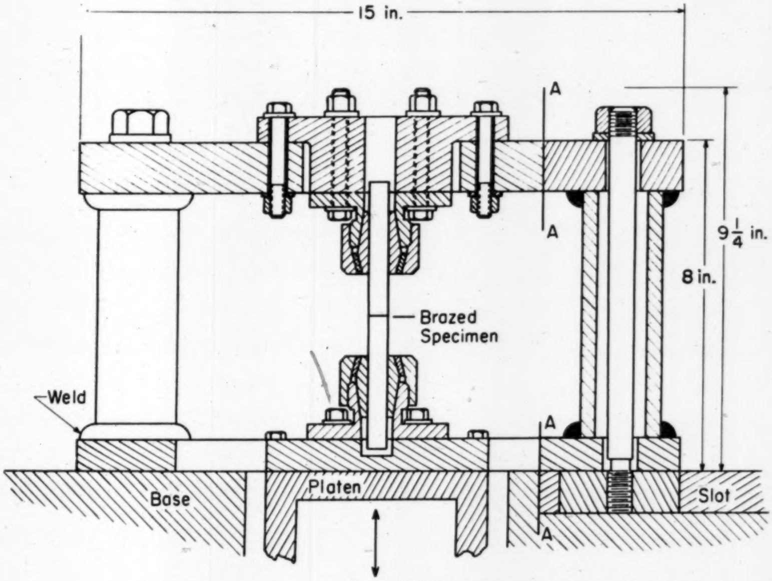

15 in.
A
A
9 1/4 in.
8 in.
Brazed Specimen
Weld
A
Base
Platen
Slot
A

FIG. 3.—Concentric Loading Fatigue Fixture.

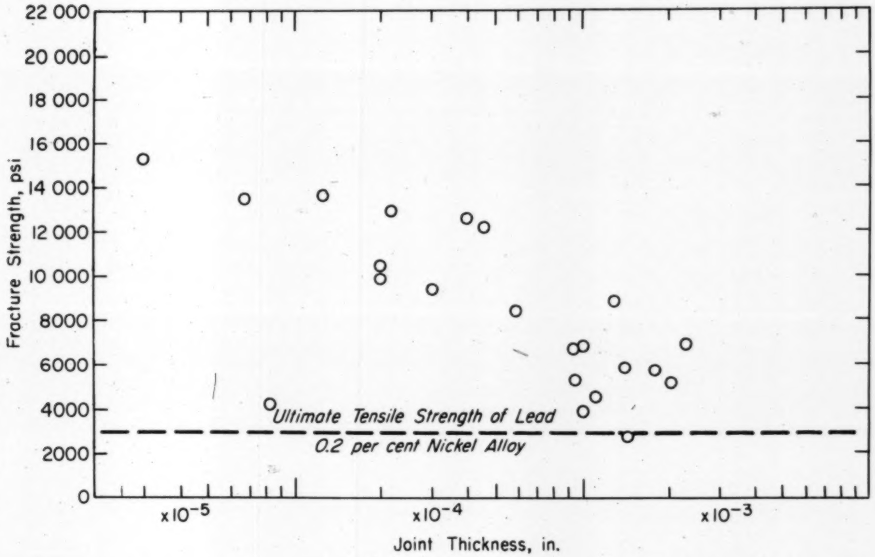


FIG. 4.—Tensile Strength of ½-in. Diameter Lead Brazed Butt Joints in 1018 Steel versus Joint Thickness

Lipsitt and Horne (12) to yield axial fluctuating tensile loading and compression-tension loading. The fixture was used with a Sonntag SF-1U Universal Fatigue machine with a maximum of 1000 lb preload plus 1000 lb dynamic load. Figure 3 is a schematic illustration of the fixture. Fabrication and alignment procedures were those as outlined by Lipsitt and Horne (12), with further details to be found in the authors' previous work (8).

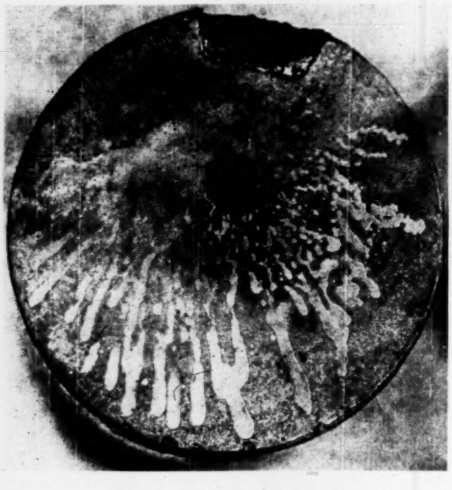


FIG. 5.—Elongated Fingerlike Voids Formed in a Shimless Lead Brazed Joint (× 5).

TENSILE PROPERTIES OF LEAD JOINTS IN MILD STEEL

Considerable effort was expended in attempting to make completely sound and voidless joints in mild steel with a filler metal of lead containing 0.2 per cent nickel to impart wettability. The effort was unsuccessful; practically no voidless joints were obtained, and the effort was abandoned.

The presence of voids is undesirable for two principal reasons: (1) the stress distribution within the filler metal disk is disturbed during tensile loading, and (2) electrolytic stripping of a joint containing voids yields an erroneous value of joint thickness. Nevertheless, the data obtained are presented in Fig. 4; and, although they are not considered completely valid because of the voids, they do indicate increasing strength with decreasing joint thickness. The highest strength obtained, over 15,000 psi at a joint thickness of 3×10^{-5} in., is five times the ultimate strength of the filler alloy in bulk.

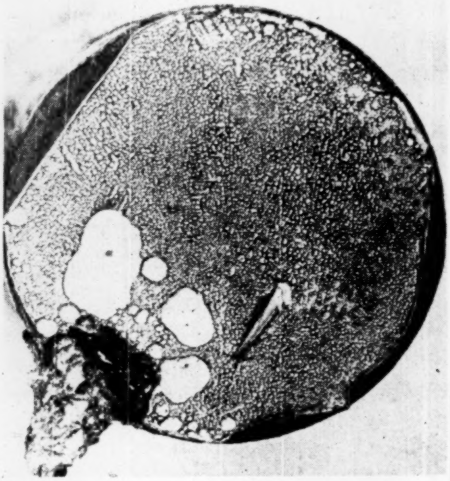


FIG. 6.—Round, Smooth-edged Voids Occurring in Shimmed Lead Joints (× 5).

The voids occurring in the filler metal during brazing were manifest in two distinct configurations, each occurring under unique circumstances. Figures 5 and 6 are photomacrographs of fractured, brazed joints showing the two void forms. The elongated finger-like voids were found only in those joints prepared without spacer shims (Fig. 5). The second form, that of round, smooth-edge voids, was found when a spacer shim was used (Fig. 6). The voids were not caused by the dewetting of the steel by the lead at the high brazing temperature. The void surfaces all show the steel to be wetted with lead. Figure 6 illustrates the wetted surface appearance of a void as compared to the nonwetted steel area at the periphery, which was restricted from being wet by the presence of the spacer wire.

The cause of void formation appears to be associated with hydrogen. Liquid lead will hold an appreciable amount of hydrogen in solution, but no hydrogen is soluble in the solid lead (**13,14**). This particular set of circumstances apparently leads to a relatively large gaseous evolution upon solidification of the lead filler alloy in the brazed joint. It is now believed that the source of the voids in both cases is the same, that of entrapped gases.

Figure 7 is a photomacrograph of a 0.010-in. thick fractured joint that had no typical large gaseous voids. The fractured surface has a peculiar appearance consisting of many craters and spires, which might be discrete and individual fractures of very ductile lead grains or groups of grains.

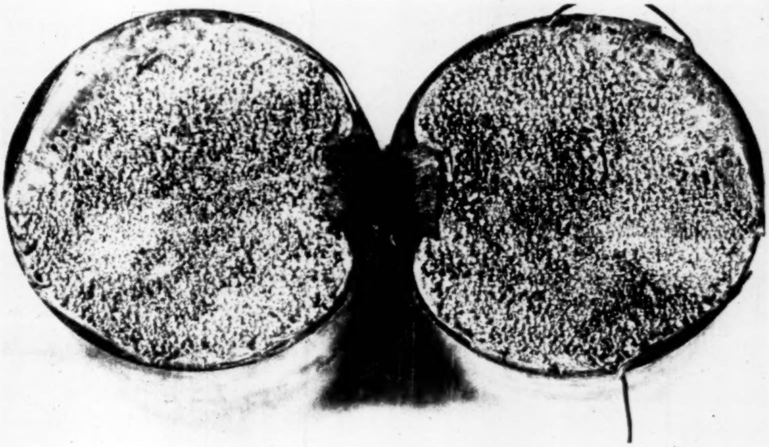


FIG. 7.—Cratered Appearance of Fractured Lead Surface in a Thick Joint (0.01 in., Unmachined) (× 5).

TENSILE PROPERTIES OF SILVER JOINTS IN 4340 STEEL

Prior to discussing the mechanical properties of the silver joints, consideration of joint quality is appropriate. Three distinct types of joint defects were encountered and had to be eliminated to provide valid data on which to base a strength-joint thickness relationship. These defects were: (1) a center void in a very thin joint, (2) a discontinuous interfacial failure occurring in the very thick joints, and (3) a shrinkage pipe in medium to thick joints.

After numerous variations and continuous refinements in the brazing procedure, these three general types of fault were virtually eliminated. Provision of an ample molten silver reservoir eliminated the shrinkage pipe; use of extremely pure, dry hydrogen eliminated the interfacial failures; and in conjunction with these a carefully controlled modified heating cycle eliminated the most serious void, the center void in the extremely thin joints. Excellent results were obtained using a converter of variable power (frequency constant) as the induction heat source. By initially heating the half-bars at extremely low power and increasing the power in carefully calibrated steps and by slowly reducing the power after flow, the proper thermal gradient for elimination of the center void is established. With this technique, perfectly sound and voidless joints were consistently obtained in the latter stages of this work.

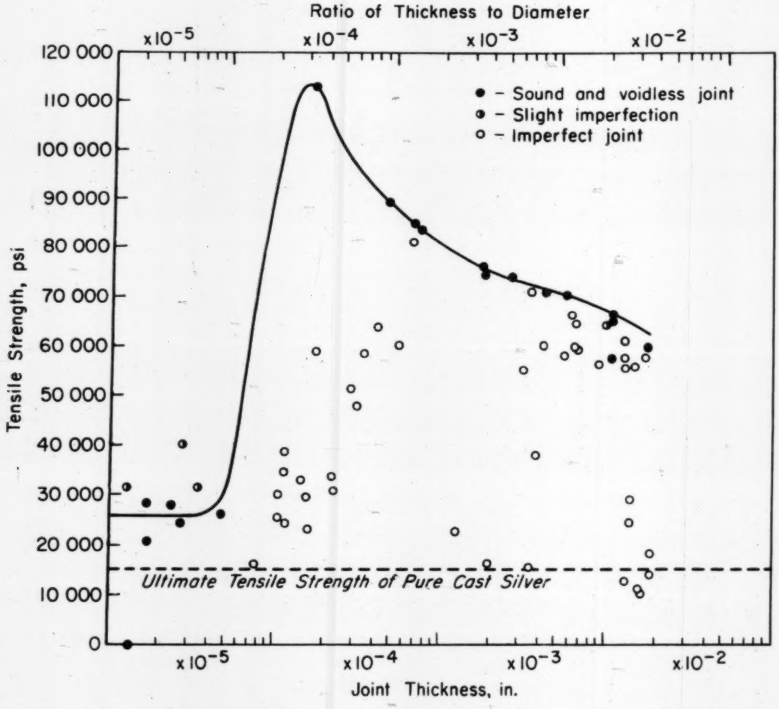


FIG. 8.—Tensile Strength of ½-in. Diameter Silver-Brazed Butt Joints in 4340 Steel versus Joint Thickness.

The mechanical data have been plotted in Fig. 8. Although all the data have been plotted, only those taken from brazes that upon visual examination show no voids or other defects have been used as reference points for the curve. These data are indicated by the solid circles. Those data indicated by the half-filled circles are from marginal brazes. An additional abscissa is used in the plot, that of ratio of joint thickness to diameter. It has been shown by Moffatt and Wulff (4) that the tensile strength of a silver-brazed joint depends not only upon the thickness but is also a linear function of the ratio of thickness to diameter. Although the diameter of all tension bars tested was constant at 0.505 in., this additional abscissa is included for possible use in future work. Inasmuch as the fatigue studies of this program were conducted on a bar of 0.250 in. in diameter, any relationships that may arise between tensile and fatigue results in this work or future work should be considered from the standpoint of ratio of joint thickness to bar diameter (t/D).

Included in Fig. 8 is a horizontal

dotted line representing the ultimate tensile strength of pure (99.95 per cent) cast silver, 15,000 psi. This datum resulted from the values obtained through the efforts of members of the staff of Wright Air Development Center.[3] Specimens were cast into $\frac{1}{2}$-in. diameter billets at 1900 F, air-cooled in the furnace and machined into tension specimens. The value of 15,000 psi as the ultimate tensile strength is an average of two bars. These silver tension bars were found to have a reduction in area of 90 per cent and an elongation of 50 per cent at fracture.

It should be cautioned, however, that the values obtained for the bulk silver are a function of cooling rate and grain size and in all likelihood have little significance if compared with the properties of silver in a brazed joint where the brazing conditions are quite different from the casting in bulk conditions.

The curve exhibits a rather sharp maximum in the region of 1×10^{-4} in. of joint thickness, attaining a strength in excess of 110,000 psi. Joints made with silver disks thinner than 1×10^{-4} in., however, exhibited low strength which, below 4×10^{-5} in., is considered constant at 26,000 psi. On the other hand, as the joint thickness increased from 1×10^{-4} in., a decrease in strength was evident, at a decreasing rate up to joint thicknesses of 7 to 8×10^{-3} in., and at an increasing rate above 7 to 8×10^{-3} in. That the inflection in the curve at joint thicknesses of 7 to 8×10^{-3} in. is real is borne out by an extrapolation of the curve established between joint thickness of 1×10^{-4} to 8×10^{-3} in. thick. If the curve did not reverse in slope, a strength in excess of 60,000 psi would have to be accepted as the strength of the infinitely thick joint, whereas the bulk strength of silver is about 15,000 psi. On this basis a reversal of the curve appears quite necessary and valid as shown by the data.

Comparing the joint thickness - tensile strength curve resulting from this work with the work of other investigators leads to interesting observations. Although the t/D values in this study (3.5×10^{-2} to 2.5×10^{-5}) overlapped those of Moffatt and Wulff (4) (2.6×10^{-1} to 1×10^{-3}) in a narrow range, strength values at comparable thicknesses cannot be compared due to widely divergent mechanical properties of the base metals used. The straight-line relationship of t/D to strength of Moffatt and Wulff was examined and not verified. The relationship was checked over a range of values from the inflection point mentioned above at $t = 7$ to 8×10^{-3} in. to the maximum strength obtained at 1.5×10^{-4} in. A plot of t/D versus tensile strength through this range yields a hyperbolic curve.

Investigating far greater thicknesses than those in this work, Bühler et al (6), when plotting joint thickness versus the log of the tensile strength, obtained straight-line relationships, and for short increments only. Bühler recognized sharp inflections in the resulting plots of his data. These inflections, unfortunately, are outside the range of joint thicknesses studied in this work. Even though silver was not examined as a filler metal, Bühler implies, however, the occurrences of the same inflection points in other metals than the lead, tin, and iron filler metals investigated. Because joint thicknesses were not made below 0.001 mm per sq mm Bühler extrapolated his curve to zero thickness and concluded the maximum strength at zero width to be the cohesive strength of the layer materials. The sharp drop in strength shown in this work makes the conclusion arrived at by Bühler untenable for real systems. Fur-

---

[3] W. J. Trapp et al, Unpublished data, Creep, Rupture and Fatigue Section, Metal Branch, Materials Laboratory, *WCLTL-5*, Wright Air Development Center, Ohio.

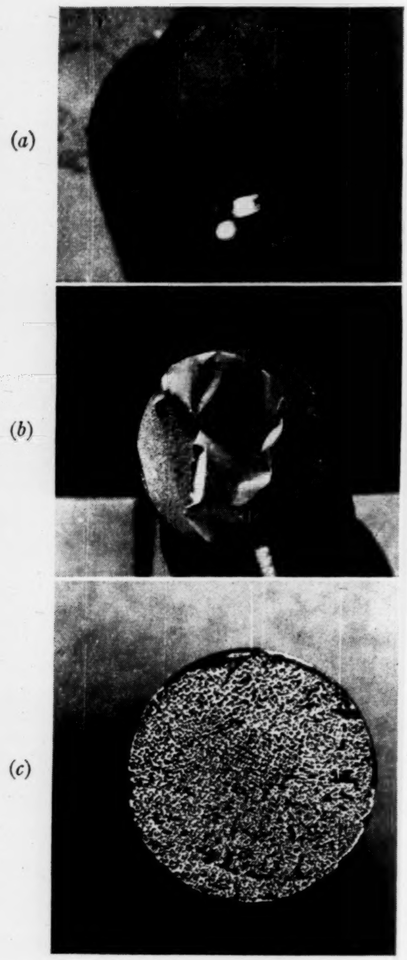


FIG. 9.—Tensile Fracture Surfaces of Brazements in 4340 Steel with Silver Filler Metal.

(a) Joint thicknesses less than 1×10^{-4} in. $\sim \times 2$
Specimen No. K4
Joint thickness = 4.65×10^{-5} in.
Tensile strength = 25, 900 psi.

(b) Joint thicknesses of the order of 10^{-4} in. $\sim \times 2$
Specimen No. K6
Joint thickness = 7.5×10^{-4} in.
Tensile strength = 83, 250 psi.

(c) Joint thicknesses greater than 2×10^{-3} in.
Specimen No. A35 $\sim \times 3$
Joint thickness = 5.70×10^{-3} in.
Tensile strength = 70. 156 psi.

ther, the expression derived by Yoshida resulting in a smooth curve does not conform to the experimental fact of the behavior of real materials. Factors, other than mechanical, affecting the shape of the curve will be discussed in later paragraphs.

Examination of the fractured surfaces of the silver disks reveals three distinct types that are coincident with the three portions of the curve, that is, very thin joints at constant strength level of 26,000 psi, medium joints where the strength decreases at a decreasing rate from a high maximum, and the very thick joints where the strength decreases more rapidly. These fracture surfaces are illustrated in Figs. 9(*a*), (*b*), and (*c*).

In those joints attaining the highest state of triaxiality, below 5×10^{-5} in. the fracture surface is perfectly smooth and uniform with near perfection of fracture symmetry through the silver disk. Representative of this is specimen K4 in Fig. 9(*a*).

The coarse grain size of the silver and the radial pattern in which the silver grains solidified can also be seen in Fig. 9(*a*).

The medium thickness joints, in which the highest strength was attained, exhibit a peculiar fracture appearance, illustrated in Fig. 9(*b*). "Leaves" of silver are attached to either face of the fracture. These leaves were extremely stiff and tough, as though having undergone unusually heavy work hardening. The size of the leaf increased, then rapidly disappeared as joint thickness increased and strength decreased. Simultaneously, the balance of the fracture surface became coarser until the leaf was replaced by a very coarse fracture and plastic contraction of the silver disk was observed. This coarse structure is the same as that observed in lead joints referred to as "crater and spire" and occurs at approximately the same joint thicknesses. This evolution of fracture appearance is shown in Fig. 9. With the leaf appearance the strength tends to level off until gross plastic deformation occurs in the silver with its associated crater and spire fracture surface. At this point the curve reverses and strength drops rapidly. No evidence of plastic deformation in the 4340 base metal was observed at any joint thickness.

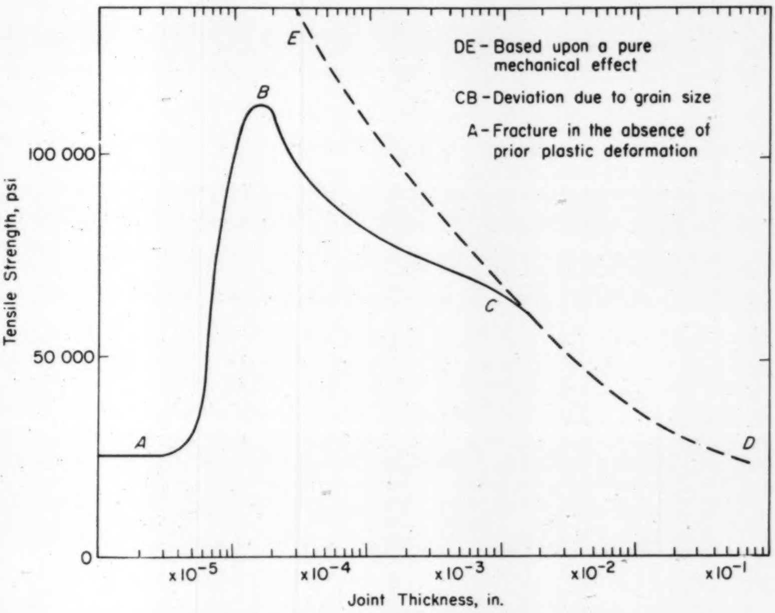


FIG. 10.—Tensile Strength - Joint Thickness Relationship.

It is interesting to speculate on the factors determining the tensile strength - joint thickness relationship in Fig. 8. For purposes of discussion, the curve is reproduced as the solid line in Fig. 10, along with dotted extrapolations to be subsequently discussed.

The most important factor in increasing the strength of silver as joint thickness decreases is the triaxiality set up by the difference in yield strength between filler and base metal. Triaxiality would reasonably be expected to increase continuously as joint thickness decreases. The fracture surfaces of the specimens in the *C* region of thickness exhibit a crater and spire appearance and a gross plastic contraction of the silver, which is postulated to be associated with relatively fine-grained silver. If the inflection at *C* is real, as seems plausible, then the extrapolation *CD* is a reasonable relationship of the pure effect of triaxiality, there being no other complicating factors in this region of thickness. If *CD* is reasonable, then an extrapolation of the pure triaxiality effect to smaller joint thickness yields *EC* which lies higher than the experimentally observed curve *BC*. How can one account for the difference between these two curves?

The difference may be due to a grain size effect. As discussed above, the fractures in the region from *B* to *C* exhibit the peculiar leaf appearance, and the leaves are apparently work hardened although no gross plastic contraction of the silver disk is evident. It is known that the grain size in the *A* region is extremely coarse, and it is believed that it is similarly coarse in the *B* to *C* region. One might therefore expect lower strength than if the grain size were relatively fine, all other things being equal. It is possible that the leaf surfaces are single crystallographic fracture planes, fracture having occurred after considerable plastic slip.

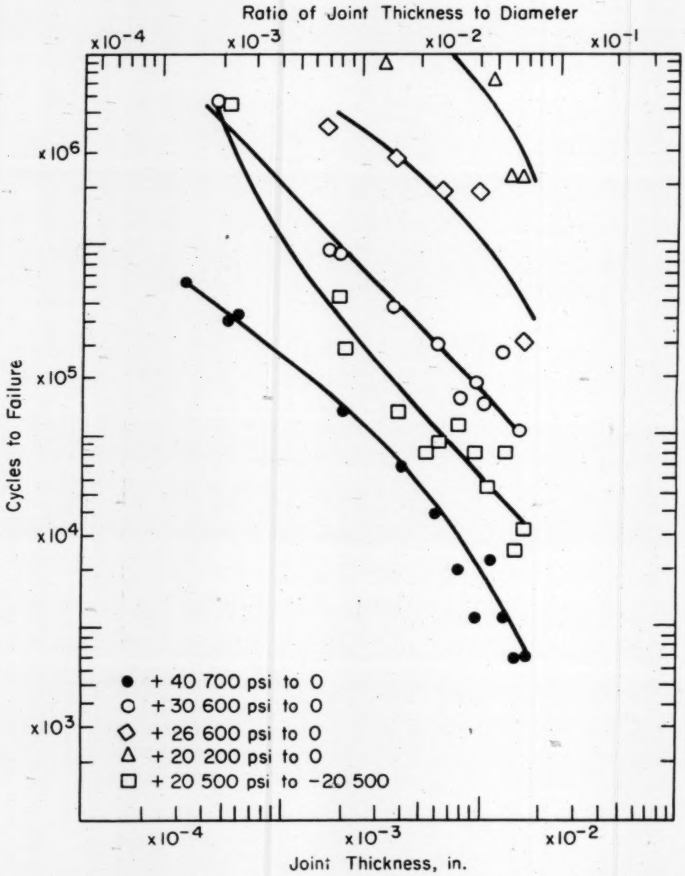


FIG. 11.—Fatigue Properties of $\frac{1}{4}$-in. Diameter Silver Butt Joints in 4340 Steel.

It is postulated that the mode of fracture in the *A* region is basically different from that in the *BC* region. In this region triaxiality may have been great enough that the silver fractured in triaxial tension without prior plastic deformation. Although to the authors' knowledge no brittle fracture has ever been observed without some prior plastic deformation, it may be conceivable that one of the principal objectives, that of having determined the brittle fracture strength of a material in triaxial tension, was achieved in the *A* portion of the curve.

Of course, the above postulations are presented on limited though fairly accurate data, and additional investigations utilizing X-ray analysis, electron microscopy, heat treatment of brazements, etc., would shed much light on the mechanisms involved.

The investigation of joints thinner than were here employed might reveal additional valuable information.

From the fluctuating tension data presented in Fig. 11, *S-N* curves were plotted using as a parameter the joint thickness to diameter ratio. These curves are presented in Fig. 12. Also included in Fig. 12 is the *S-N* curve for pure silver. These data were obtained from bars prepared in the manner described previously by workers at Wright Air Development Center.[3] Again, the relationship between the fatigue strength of pure bulk silver and the silver in the brazed joint is obscured by numerous metallurgical factors, and caution is advised in an attempt to establish any relationship. As the fatigue strength was determined for only one push-pull stress level, similar push-pull *S-N* curves could not be plotted.

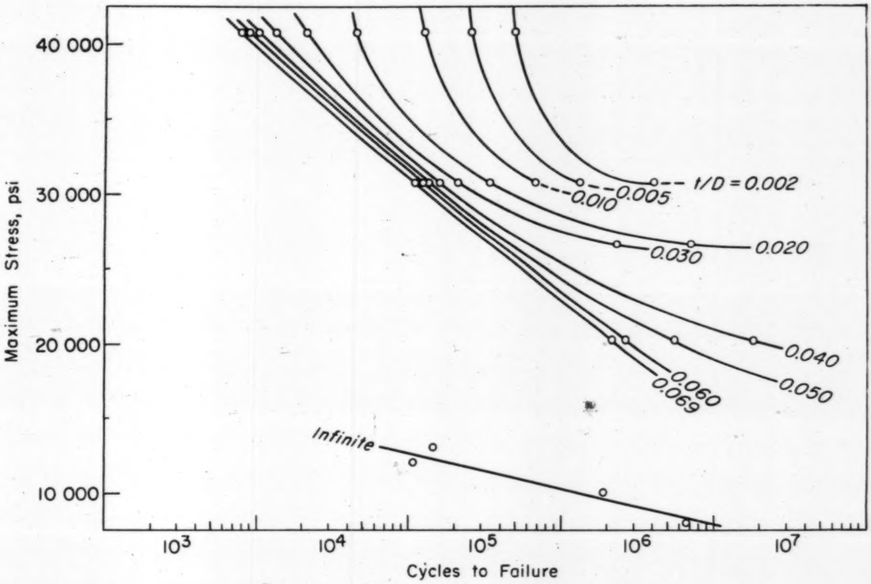
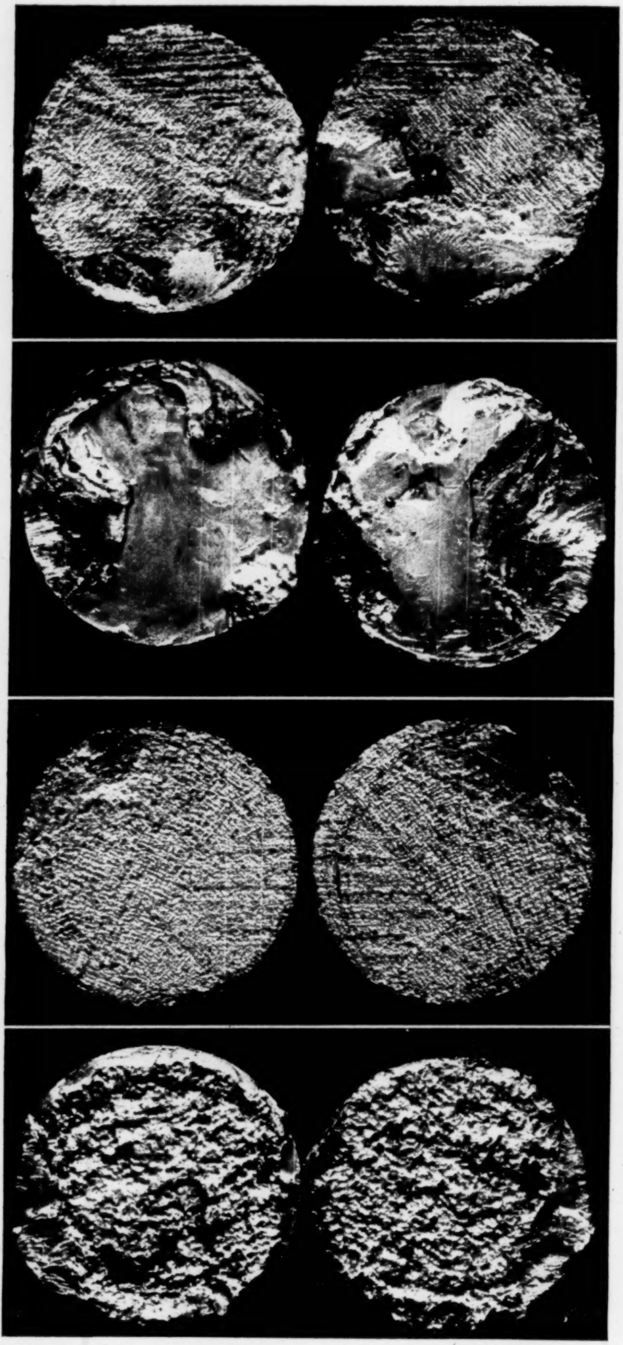


FIG. 12.—Fluctuating Tension *S-N* Curves of $\frac{1}{4}$-in. Diameter Silver-Brazed Butt Joints in 4340 Steel as a Function of Joint Thickness to Diameter Ratio.

Stress cycled between 0 and the value on the ordinate scale.

Typical fracture surfaces of fatigue-tested specimens are illustrated in Fig. 13. In general, the appearance of the fracture surface fell into one of several types. No positive correlation could be observed between type of fracture surface and stress direction or magnitude, joint thickness, or number of cycles to failure. Specimens K40 and K38, both of relatively small joint thickness, exhibit a regularity of strain markings which possibly indicate very large grains. In addition, for both these specimens the point of origin of the fatigue failure can be seen at the surface of the bars and at the base metal - filler metal interface. Although the pattern has suffered in photography and reproduction, lines were observed in the silver extending



Specimen No. K40
Joint thickness 3.88 × 10^{-3} in.
Loaded ± 20, 500 psi
131, 000 cycles to failure.

Specimen No. K68
Joint thickness 1.34 × 10^{-2} in.
Loaded ± 20, 500 psi
79, 000 cycles to failure.

Specimen No. K38
Joint thickness 3.73 × 10^{-3} in.
Loaded + 30, 600 psi
458, 000 cycles to failure.

Specimen No. K61
Joint thickness 1.15 × 10^{-2} in.
Loaded + 40, 700 psi
22, 000 cycles to failure.

FIG. 13.—Typical Fracture Surfaces of Fatigue-Tested Silver-Brazed Butt Joints in 4340 Steel (× 6).

from the fracture origin for a short distance into the silver. Although the fatigue fracture originated at the base metal - filler metal interface, the fracture propagated principally symmetrically through silver.

Specimen K68 is an example of a specimen with a relatively thick joint which fractured primarily at the base metal - filler metal interface. However, even in this case bare steel was not visible; some silver remained on the steel surfaces after fracture. This type of fracture is therefore not associated with any lack of wetting during brazing.

Specimen K61 also had a relatively thick joint, and fracture occurred roughly in the plane of symmetry of the silver disk. The fracture appearance is somewhat similar to the crater and spire fractures observed in thick joints loaded in static tension.

SUMMARY AND CONCLUSIONS

Experimental procedures and equipment have been developed for making sound and voidless brazed joints of silver filler metal in 4340 steel and for tension and fatigue testing such joints to provide data on the effect of triaxial stresses on fracture.

In regard to the static tensile properties of silver butt brazements in 4340 steel, the following observations and conclusions may be made: Over the range of joint thickness from 2×10^{-2} in. to 1.5×10^{-4} in., joint strength increases with decreasing thickness due to the increasing triaxiality of stress. An inflection in the strength-thickness curve is observed at joint thicknesses of 7 to 8×10^{-3} in. and, based upon the appearance of the fracture surfaces, it is postulated that the inflection is due to a grain size effect. At joint thicknesses above 7 to 8×10^{-3} in. the silver is relatively fine grained, the fracture surface consists of multitudinous craters and spires, and gross plastic contraction of the silver disk occurs. At joint thicknesses between 7 to 8×10^{-3} in. and 1.5×10^{-4} in., the fracture surface consists of cold-worked leaves which may be single crystals, and no gross plastic contraction of the silver disk is observed. The maximum in the tensile strength - joint thickness curve occurs at 1.5×10^{-4} in. where a strength greater than 110,000 psi is obtained, some $7\frac{1}{2}$ times the strength of silver in uniaxial tension. At joint thicknesses less than 4×10^{-5} in. strength is relatively constant at 26,000 psi, the silver grains are large and sometimes form a radial pattern in the disk, and no evidence of plastic deformation is observed in the silver or base metal. It is postulated that this might be the brittle fracture strength of silver in the absence of prior plastic flow, a condition achieved in these tests by the triaxial tension existing in the extremely thin silver disk.

The fatigue life of silver-brazed butt joints in 4340 steel increases continuously with decreasing joint thickness for both completely reversed axial loading and fluctuating tensile loading over the entire range of joint thickness investigated, 3×10^{-4} in. to 1.8×10^{-2} in.

Acknowledgments:

This program was sponsored by the Air Force through the Materials Laboratory, Wright Air Development Center. Appreciation is expressed to the Air Force for permission to publish, and to W. J. Trapp and his colleagues at Wright Air Development Center for their encouragement and assistance.

REFERENCES

(1) E. Orowan, "Notch Brittleness and the Strength of Metals," *Transactions*, Inst. of Engineers and Shipbuilders in Scotland, Vol. 89, pp. 165–204 (1946).

(2) N. Bredzs, "Investigation of Factors Determining the Tensile Strength of Brazed Joints," *Welding Journal* (Research Supplement), Vol. 33, No. 11, Nov. 1954, pp. 545s–563s.

(3) N. Bredzs and H. Schwartzbart, "Triaxial Tension Testing and the Brittle Fracture Strength of Metals," *Welding Journal* (Research Supplement), Vol. 12, No. 12, Dec. 1956, pp. 610s–615s.

(4) W. G. Moffatt and J. Wulff, "Strength of Silver-Brazed Joints in Mild Steel," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 209, p. 442 (1957).

(5) T. Yoshida, "Strength of Brazed Joint," *Journal of the Japan Welding Soc.*, Vol. 26, No. 11, pp. 46–50 (1957).

(6) H. Bühler and J. Colbus, "Die Festigkeitsstergerung von Spaltlötverbindung bei abnehmender Spaltbreite als Erscheinung der Festigkeit bei behinderter Verformung," *Zeitschrift für Metallkunde*, Vol. 48, No. 2, pp. 66–71 (1957).

(7) G. L. J. Bailey and H. C. Watkins, "The Flow of Liquid Metals on Solid Metal Surfaces and Its Relation to Soldering, Brazing and Hot Dip Coating," *Journal of the Institute of Metals*, Vol. 80, Part 2, Oct. 1951, pp. 57–76.

(8) W. Lehrer and H. Schwartzbart, "Investigation of Static and Fatigue Strength of Metals Subjected to Triaxial Stresses," *WADC Technical Report*, No. 58-430, Wright Air Development Center, Dec., 1957.

(9) H. T. Francis, "An Electrolytic Thickness Tester for Plated Metal Coatings," *Journal of the Electrochemical Soc.*, Vol. 93, No. 3, pp. 79–83 (1948).

(10) G. Sachs, J. D. Lubahn, and L. J. Ebert, "Notched Bar Tensile Test Characteristics of Heat-Treated Low Alloy Steels," *Transactions*, Am. Soc. for Metals, Vol. 33, pp. 340–391 (1944).

(11) W. F. Brown, Jr., H. Schwartzbart, and M. H. Jones, "Tensile-Fracturing Characteristics of Several High Temperature Alloys as Influenced by Orientation with Respect to Forging Direction," *NACA Research Memorandum*, Nat. Advisory Committee for Aeronautics, No. E50L28, Feb. 12, 1947.

(12) H. A. Lipsitt and G. T. Horne, "Investigation of the Fundamentals of Fatigue in Metals," Ph.D. Thesis, under *NACA Contract No. NAW-6407*, Metals Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Pa.

(13) W. R. Opie and N. J. Grant, "Solubility of Hydrogen in Molten Lead," *Journal of Metals*, Vol. 3, No. 3, pp. 244–245 (1951).

(14) W. Mannchen and M. Baumann, "Absorption of Hydrogen by Lead and Alloys of Lead with Mg and Ca," *Metall*, Vol. 9, No. 15/16, pp. 686–688 (1955).

RELATION BETWEEN ROLLING-CONTACT FATIGUE LIFE AND MECHANICAL PROPERTIES FOR SEVERAL AIRCRAFT BEARING STEELS*

By E. V. Zaretsky[1] and W. J. Anderson[1]

Synopsis

Two bench-type fatigue rigs were used to determine the room-temperature fatigue lives of groups of AISI M-1, AISI M-50, Halmo, and WB-49 tool-steel balls, tempered to various hardness levels. Ten per cent life increased continuously with increasing hardness. In contrast, elastic-limit and yield-strength measurements made on bar specimens from the same heats of materials showed optimum values at an intermediate hardness level and so did not correlate with fatigue life. Resistance to plastic deformation, measured with ball specimens in rolling contact, did correlate with fatigue life.

The determination of rolling-contact fatigue life of a particular bearing steel is an expensive undertaking, because the very high scatter in fatigue lives makes it necessary to test a large number of specimens. The cost is high both in money and time. Therefore, a reliable means of predicting relative fatigue life without actually testing large numbers of specimens would be a valuable achievement. For this reason, much effort is being spent on relating various material properties to the fatigue life of bearing elements. Any correlation between an easily measured property of bearing materials and rolling-contact fatigue life would be very helpful in screening potential bearing materials. It would be logical to determine whether a correlation exists between fatigue life and such easily measured properties as elastic limit, yield strength, ultimate strength, and resistance to impression or hardness.

One of the key factors in determining whether a correlation exists between one of these easily measured properties and fatigue life is the rate of strain occurring in the tests for those properties. Although the rate of strain used in determining both elastic limit and hardness is low (compared with that encountered in high-speed rolling-contact bearings), the rate used in hardness tests is somewhat higher than that used in tests for elastic limit. Furthermore, the stress pattern generated by the contact of the hardness indenter is more similar to that of bodies in rolling contact than is the stress pattern in a tension or compression test specimen. For these reasons, hardness may be a measurable material property that correlates with fatigue.

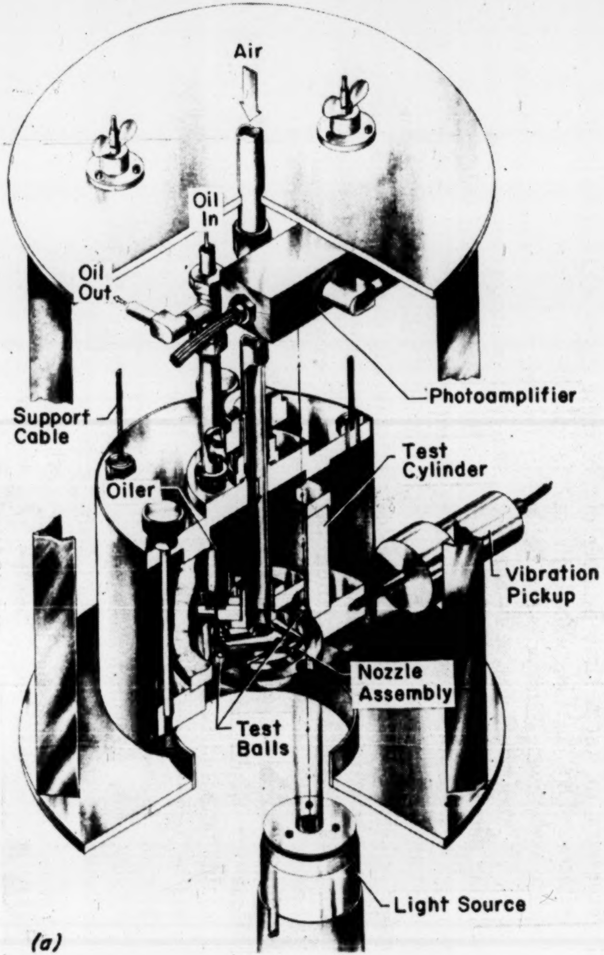
Some fatigue data have been obtained which indicate that fatigue life varies with material hardness. Jackson (**1**)[2] has

---

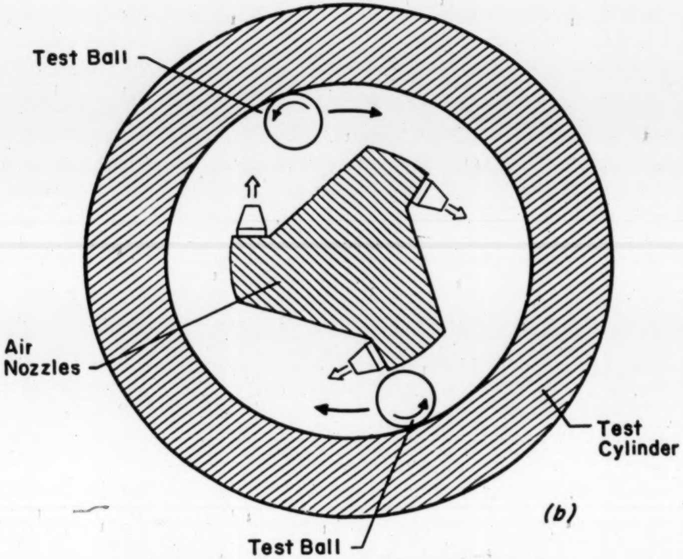
* Presented at the Sixty-third Annual Meeting at the Society, June 26–July 1, 1960.

[1] Lewis Research Center, National Aeronautics and Space Administration, Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

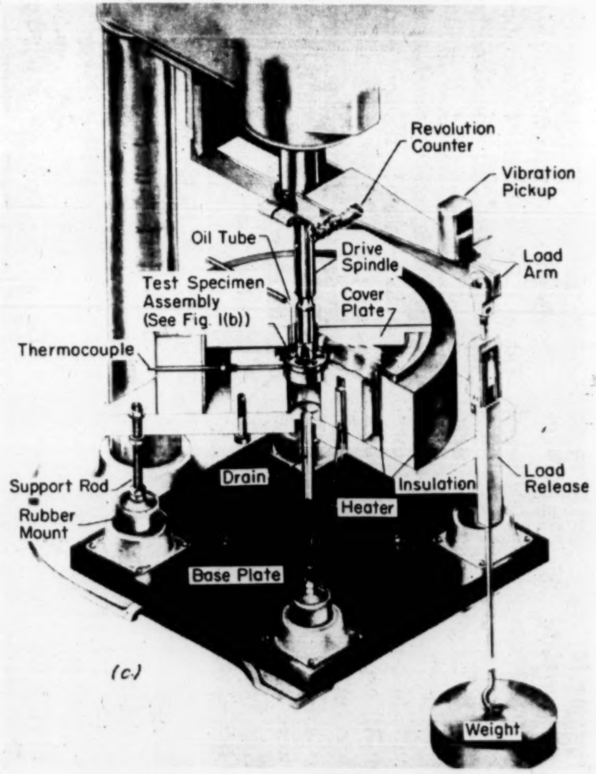

Air
Oil In
Oil Out
Photoamplifier
Support Cable
Test Cylinder
Oiler
Vibration Pickup
Nozzle Assembly
Test Balls
Light Source
(a)

(a) Cutaway view, rolling-contact fatigue spin rig.

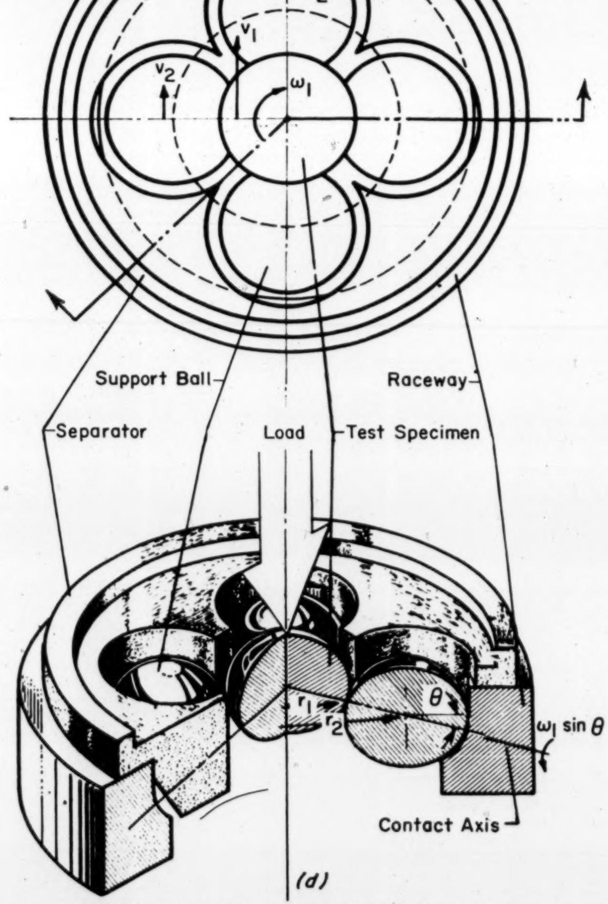

Test Ball
Air Nozzles
Test Cylinder
Test Ball
(b)

(b) Schematic diagram, rolling-contact fatigue spin rig.

D


Revolution Counter
Vibration Pickup
Oil Tube
Drive Spindle
Load Arm
Test Specimen Assembly (See Fig. 1(b))
Cover Plate
Thermocouple
Support Rod
Rubber Mount
Drain
Heater
Insulation
Load Release
Base Plate
Weight
(c)

(*c*) Five-ball fatigue tester.


ω2
v1
v2
ω1
Support Ball
Raceway
Separator
Load
Test Specimen
θ
r1
r2
ω1 sin θ
Contact Axis
(d)

(*d*) Schematic diagram, five-ball fatigue tester.

FIG. 1.—Test Apparatus.

shown that single-ball fatigue tests show an increase in life with higher hardness. In addition, unpublished full-scale bearing data have shown that rolling-contact fatigue life improves with increasing material hardness. However, none of these data included the ultimate hardness obtainable for the materials used. Some experimental work done on the determination of mechanical strength properties of hard steels by Muir et al (**2**) has shown that, for the materials tested, mechanical strength increases with hardness to a maximum and then decreases at very high hardness values. This suggests that the maximum rolling-contact fatigue life for a given alloy composition may also be obtained at a hardness level below the maximum obtainable for that material. This is, of course, based on the premise that a correlation between fatigue life and some of these measured mechanical properties exists.

The object of this research was to obtain rolling-contact fatigue data for several tool steels over a range of hardness levels for each steel and to attempt to correlate these data with mechanical properties such as resistance to plastic deformation and wear, elastic limit, yield strength, and ultimate strength in both tension and compression, and also with per cent retained austenite. All experimental results for a given alloy composition were obtained from the same heat of material. All failures were obtained with ball specimens. This paper is based on work reported initially by Carter et al (**3**).

APPARATUS

Two bench-type fatigue rigs were used. The first of these, the fatigue spin rig, is shown schematically in Figs. 1(*a*) and (*b*) and has been described in detail by Macks (**4**). Essentially, it consists of two balls driven at high speed on the inner surface of a race cylinder by an air jet (Fig. 1(*b*)). Loading is supplied by centrifugal force. Instrumentation provides for speed control and automatic failure detection and shutdown when a fatigue spall occurs.

The second type of bench rig, the five-ball fatigue tester, is shown in Figs. 1(*c*) and (*d*). It consists essentially of a driven test ball pyramided upon four lower balls, which are positioned by a separator and free to rotate in an angular-contact raceway. Specimen loading and drive is supplied through a vertical shaft. By varying the pitch diameter of the four lower balls, the bearing contact angle, θ, (Fig. 1(*d*)) may be controlled. Again by use of a failure detection and shutdown system, long-term unmonitored tests can be run. In both of these rigs, the test balls were lubricated by a synthetic diester fluid meeting the MIL-L-7808 specification introduced in droplet form.

TEST SPECIMENS

Balls $\frac{9}{16}$ in. in diameter of M-1, M-50, and Halmo tool steels, having the nominal chemical compositions shown in Table I, were used. These specimens were fabricated from the same heats of material that were used to produce the bar specimens used by another investigator to establish tension and compression strengths for these alloys. This identity of material origin makes possible the direct comparisons of ball and bar tests that are discussed later. Balls $\frac{1}{2}$ in. in diameter made from WB-49 tool steel were also tested. The composition of this material is also given in Table I. Details of the heat-treatment schedules, which produced a range of hardness for each of the four alloys tested, are shown in Table II. For each alloy composition, tempering temperature was the only variable in specimen preparation.

The per cent by volume of retained

TABLE I.—ANALYSIS OF TEST MATERIALS.

Material	Analysis, per cent by weight										
	Carbon	Phosphorus	Sulfur	Manganese	Silicon	Chromium	Vanadium	Tungsten	Molybdenum	Nickel	Cobalt
AISI M-1	0.80	0.004	0.007	0.25	0.32	3.76	1.15	1.53	8.54	0.07	...
AISI M-50	0.81	0.004	0.007	0.26	0.14	3.97	1.07	0.01	4.29	0.05	...
Halmo	0.59	0.005	0.007	0.31	1.10	4.79	0.51	...	5.22	...	...
WB-49	1.07	0.006	0.007	0.30	0.02	4.4	2.0	6.8	3.9	0.04	5.2

TABLE II.—TEST MATERIAL PROPERTIES AND HEAT TREATMENT.

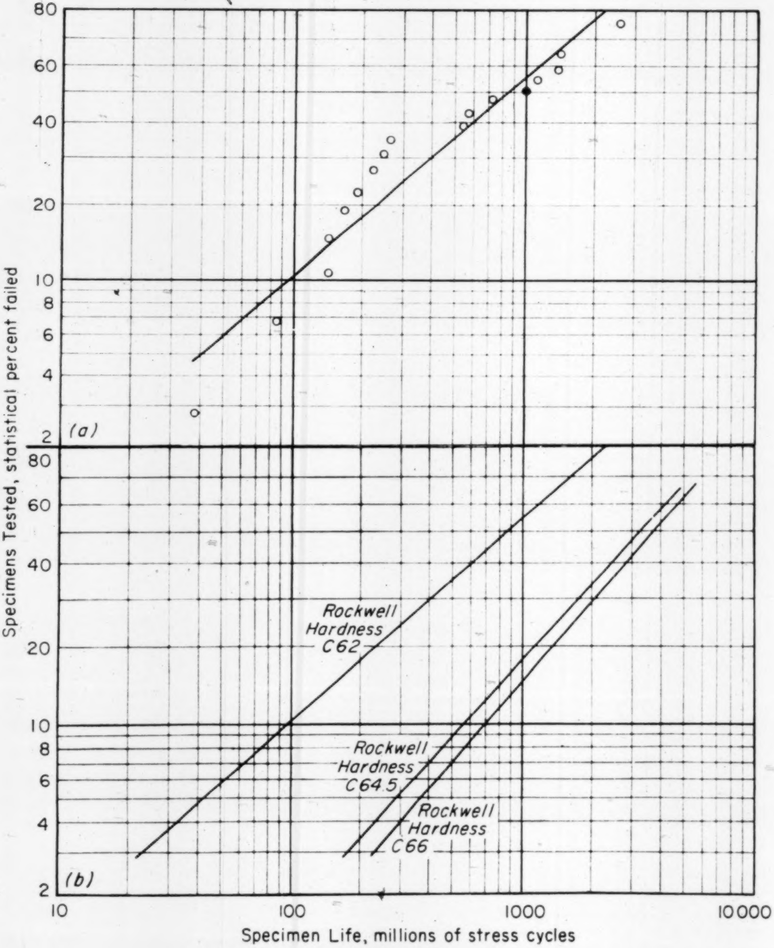
Melting process, induction vacuum.

Material	Rockwell Hardness C Scale	Retained Austenite, per cent by volume	Austenite Grain Size, ASTM Classification E 19[a]	Heat Treatment					
				Preheat, deg Fahr	Austenite, deg Fahr	Quench, deg Fahr	1st temper, 120 min, deg Fahr	2nd temper, 120 min, deg Fahr	3rd temper, 40 min, deg Fahr
AISI M-1	62	2.8	8−	salt:	2200	oil:	1100	1100	...
	64.5	3.2	8−	1450,	2200	120	1050	1050	...
	66	7.2	8−	3½ min	2200		1000	1000	...
AISI M-50	60	1.8	8−	salt:	2050	oil:	1100	1100	...
	60.5	2.2	8−	1450,	2050	120	1050	1050	...
	62	...	8−	3½ min	2050		1000	1000	1025
	63	4.2	8−		2050		1000	1000	...
Halmo	59	2.6	6 to 7	salt:	2100	oil:	1100	1100	...
	60	3.6	6 to 7	1450	2100	120	1050	1050	...
	62	4.4	6 to 7	3½ min	2100		1000	1000	...
WB-49	55	<1	8+	salt:	salt:	salt:	1025	1025	1250
	60	<1	8+	1500,	2225	1225,	1025	1025	1200
	65	<1	8+	6 min	6 min	6 min	1025	1025	1150
	68	5.0	8+				1025	1025	...

[a] Classification of Austenite Grain Size in Steels (E 19 – 46), 1958 Book of ASTM Standards, Part 3, p. 488.

austenite was determined by X-ray diffraction techniques for specimens from each of the heat treatments of the four alloys. A series of calibration specimens with a known per cent austenite was prepared by inserting austenitic stainless-steel wires in a cross-section of low-carbon (ferritic) steel. The intensity of a diffraction line characteristic of austenite was measured for the known specimens, and a calibration curve of per cent austenite against line intensity was plotted. This calibration was used to determine the per cent retained austenite for each of the samples. These data are given in Table II.

The race cylinders used in the spin rig were made from the same heats of material as the balls tested in this rig. All cylinders from the same alloy received the same fabrication and heat treatment, so that they had the same hardness (approximately Rockwell hardnesses C 62 to C 63). Thus, cylinders run with the various hardness ball groups of a given alloy were nominally identical.



(*a*) Rockwell hardness C 62.
(*b*) Summary of fatigue lives for three hardness levels.

FIG. 2.—Rolling-Contact Fatigue Life at 800,000-psi Maximum Hertz Stress of M-1 Tool-Steel Balls Tempered to Various Hardness Levels.

RESULTS AND DISCUSSION

Fatigue Life and Load Capacity:

The fatigue-life results obtained with M-1 balls of varying hardnesses are shown in Fig. 2. Rolling-contact fatigue data are usually presented on a "Weibull plot." This system of coordinates is designed to produce a straight line from any set of rolling-contact fatigue data. The ordinate scale represents the log-log of the reciprocal of the probability of survival, and the abscissa scale, the log of life in stress cycles. For convenience, the ordinate scale is graduated in per cent of specimens failed. Data points are included in Fig. 2(*a*) to illustrate the typical array of points that results when a Weibull plot is made from a set of rolling-contact fatigue data. After all of the data points have been plotted on a set of Weibull coordinates, a best-fit

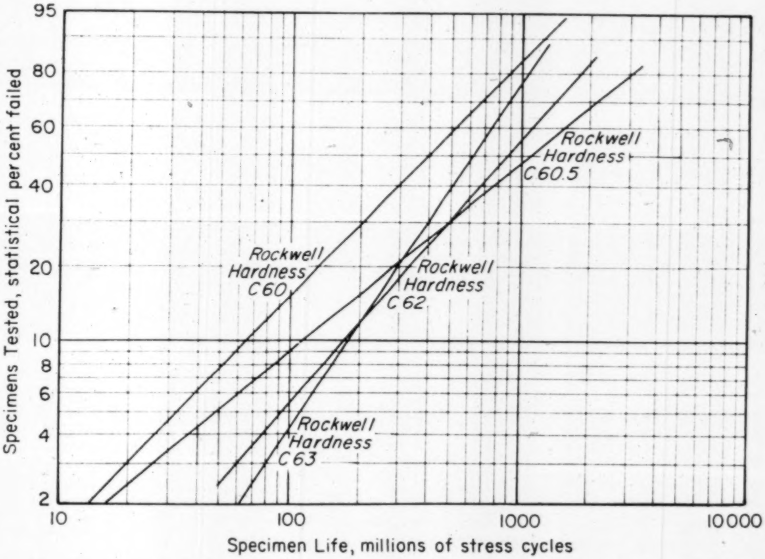


FIG. 3.—Rolling-Contact Fatigue Life at 800,000-psi Maximum Hertz Stress of M-50 Tool-Steel Balls Tempered to Various Hardness Levels.

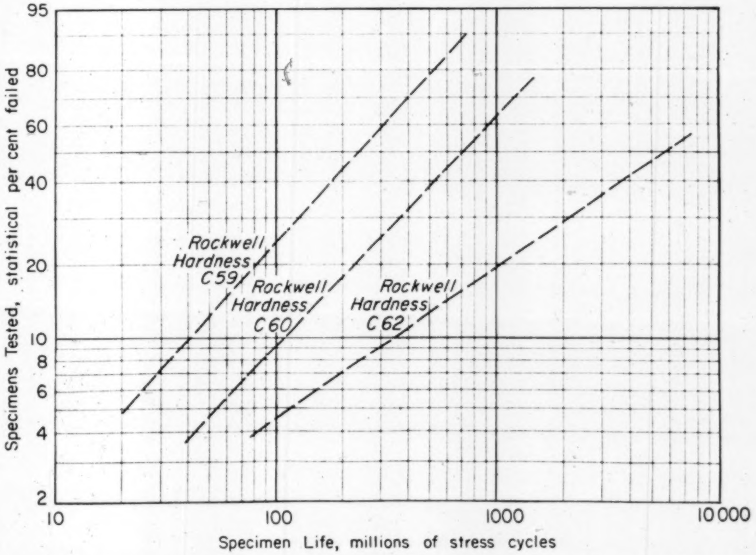


FIG. 4.—Rolling-Contact Fatigue Life at 800,000-psi Maximum Hertz Stress of Halmo Tool-Steel Balls Tempered to Various Hardness Levels (adjusted from 750,000 psi).

straight line is drawn through the array of points. From this straight line two significant fatigue lives are obtained: the lives at which 10 per cent and 50 per cent of the specimens have failed. Since in aircraft applications greater interest centers around the early probability of failure, the so-called 10 per cent life is usually the life used in analyzing these data.

Figure 2(*b*) shows the resulting Weibull lines without the data points that resulted with M-1 balls having Rockwell hardnesses of C 62, 64.5, and 66. It can be seen that life increases with increasing hardness with this material. Similar results obtained with M-50 balls are shown in Fig. 3. Here, the hardnesses tested were Rockwell C 60, C 60.5, C 62, and C 63. Again, as for the M-1 steel, a general increase in life can be seen with increasing hardness.

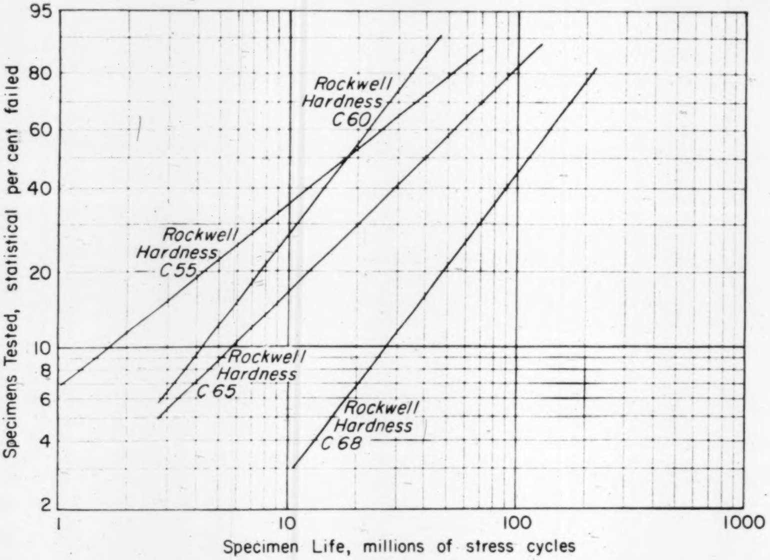


FIG. 5.—Rolling-Contact Fatigue Life at 800,000-psi Maximum Hertz Stress and 40-deg Contact Angle of WB-49 Tool-Steel Balls Tempered to Various Hardness Levels.

Figure 4 illustrates the fatigue-life results obtained with Halmo balls. Here the hardness levels tested were Rockwell hardnesses C 59, C 60, and C 62. In Fig. 4, the Weibull lines are drawn as dashed lines because the Halmo balls were tested at 750,000 psi as compared to 800,000 psi for the M-1 and M-50 balls. Therefore, the lines shown in Fig. 4 were shifted to account for the difference in test stress. The data shown in Figs. 2, 3, and 4 were all obtained in the fatigue spin rig. Detailed test data used to obtain Figs. 2, 3, and 4 are reported by Carter et al (3).

Figure 5 summarizes the fatigue-life data obtained with the WB-49 balls These data were obtained in the five-ball fatigue tester, because it was found that speed limitations for the softer balls in this group prevented attaining the speeds necessary to achieve high stresses in the fatigue spin rig. As for the previous materials, fatigue life of the WB-49 balls generally increases with increasing hardness over the range tested.

Figure 6 is a plot of the 10-per cent life against hardness for the four alloys tested. The M-1, M-50, and Halmo compositions were tested at 0-deg contact angle in the fatigue spin rig, and the WB-49 alloy at a 40-deg contact angle in the five-ball tester. This figure shows that there is a general trend toward longer fatigue life with increased material hardness.

Results for M-50 steel (Fig. 3) are in good agreement with Jackson's data (1), which show a progressive increase in fatigue life with higher material hardness. Baughman (5) has presented fatigue data obtained with M-50 steel. An analysis of these data based on a complex empirical equation predicts a maximum life at a Rockwell hardness of C 64. However, only limited data were obtained by Baughman (5) at the Rockwell hardness C 64 level.

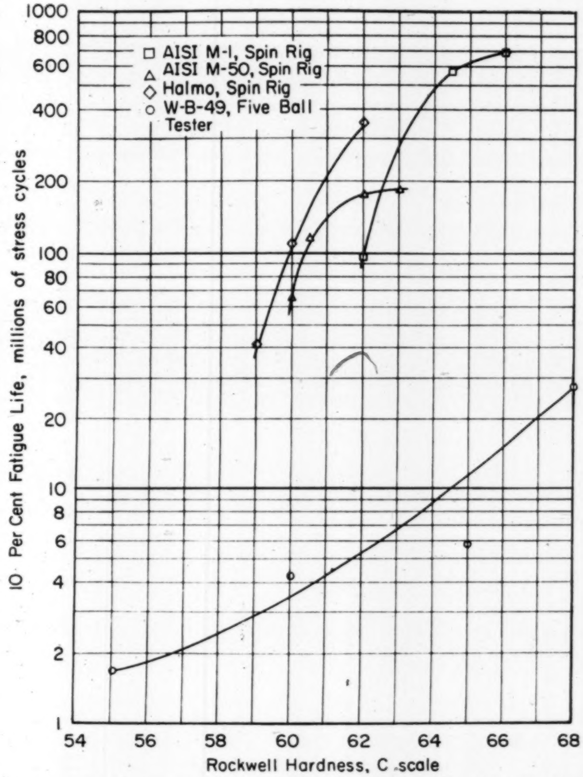


FIG. 6.—Ten per cent Fatigue Failure Life as a Function of Hardness for Four Tool Steels.

An important criterion of a bearing is its load-carrying capacity. The load-carrying capacities of the four alloys may be calculated at the various hardness levels from the fatigue-life results, summarized in Fig. 6. Load capacity, contact load, and life are related by the equation

$$C = P\sqrt[n]{L_N}$$

In this equation, C is the load capacity or the contact load, in pounds, that will produce failure of 10 per cent of the test specimens in one million stress cycles; P is the actual load, in pounds; L_N is the actual 10-per cent life in millions of stress cycles; and n is an exponent relating load and life, usually taken as 3 for bearing steels. The load capacity for each hardness level of the four alloys studied and the reductions in load capacity of softer specimens relative to the hardest in each group are given in Table III. These calculations show that a large variation in load capacity may be produced by control of material hardness within the range applicable to bearing steels and that the highest hardness for a given alloy shows the highest load capacity. It should be noted that a comparison cannot be made between materials tested in the five-ball tester and the spin rig because of the differences in the contact geometry of the two rigs.

Effect of Hardness on Mechanical Strength:

Some data are available in the literature on the variation of mechanical-strength properties such as elastic limit, yield strength, and ultimate strength in both tension and compression with material hardness. Muir et al and Grobe et al (2,6) have observed a peak in the measured strength values at hardness values approaching the ultimate attainable hardness and a subsequent loss in measured strength at higher hardness values. If one assumes that a correlation should exist between fatigue life and any of these mechanical properties, these observations suggest that a possible maximum in rolling-contact fatigue life at an intermediate hardness level close to the maximum for a particular alloy might exist.

TABLE III.—LOAD CAPACITY OF TEST MATERIALS AT VARIOUS HARDNESS LEVELS.

Material	Rockwell Hardness, C Scale	Actual Load, P, lb	Ten Per Cent Fatigue Life, L_N, millions of stress cycles (Fig. 6)	Load Capacity (Contact Load), C, lb	Ratio of C to Hardest in Group
AISI M-1 (spin rig)	62	875	97	4020	0.518
	64.5	875	570	7240	0.934
	66	875	695	7750	1.000
AISI M-50 (spin rig)	60	875	65	3520	0.711
	60.5	875	116	4270	0.862
	62	875	176	4890	0.989
	63	875	182	4950	1.000
Halmo (spin rig)	59	875	41	3070	0.498
	60	875	110	4160	0.676
	62	875	350	6160	1.000
WB-49 (five-ball tester)	55	173	1.7	206	0.394
	60	173	4.3	281	0.538
	65	173	5.8	303	0.580
	68	173	27.5	522	1.000

As part of a coordinated research effort of which the fatigue data reported here are a portion, the elastic limit, yield strength, and ultimate strength in both tension and compression at various hardness levels were obtained for bar specimens from the same material heats of M-1, M-50, and Halmo as were used to produce these rolling-contact fatigue data. These data, published by Sachs et al (7,8), show trends that are generally similar to those obtained by Muir et al and Grobe et al (2,6). In general, an increase in mechanical strength with higher hardness was observed up to an optimum hardness, after which strength tended to deteriorate at very high hardness values. There is some question as to whether this deterioration actually exists.

An apparent deterioration in strength could be produced by minor eccentricities in loading that would cause higher than nominal stresses in very hard specimens. As stated by Sachs et al (7), a deterioration in strength at very high hardness may be due to residual stresses not relieved at the low tempering temperature used to produce the highest material hardnesses. An indication that residual stresses may not be responsible for the loss in strength at very high hardness is provided by the SAE 52100 data given by Sachs et al (**7,8**). This steel shows a maximum strength at an intermediate hardness as do the tool steels measured by these authors (**7,8**). However, the rotating-beam fatigue life data for this 52100 steel given by Sachs et al (**7,8**) do not show a maximum at an intermediate hardness. Since residual stresses are in tension at the surface, and therefore additive in the rotating-beam tests, a maximum at an intermediate hardness and subsequent loss in life at the highest hardnesses would be anticipated if residual stresses of a significant magnitude were present. The fact that no loss in rotating-beam fatigue life was observed at very high hardnesses indicates that residual stresses may not be the primary cause of loss in strength of very hard bar specimens.

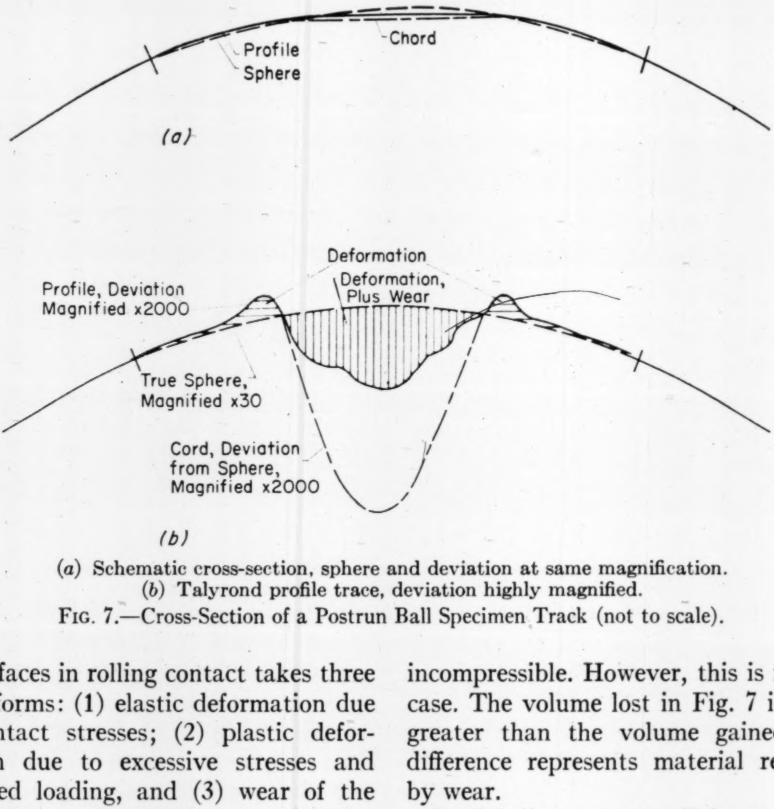
Sachs et al (**7,8**) also report an apparent decrease in elastic modulus at high hardness values. This may be further evidence of overstressing due to eccentric loading. The results obtained by Sachs et al (**7,8**) are for the same heats of material as the ball specimens tested in this investigation. Higher than nominal stresses would produce a greater amount of strain and hence reduce the ratio of nominal stress to actual strain, which is the measured elastic modulus.

From the data on mechanical properties reported by Sachs et al (**7,8**) and the fatigue-life data reported here, it can be seen that mechanical strength does not correlate with rolling-contact fatigue life. Mechanical strength was highest at an optimum intermediate hardness and somewhat lower at the highest hardness values, while fatigue life improved continuously with increasing hardness. This may be due to a true lack of correlation between mechanical strength and fatigue life, to the presence of undetected stress concentrations, or to the presence of residual stresses not removed by tempering in the hardest tension and compression test specimens. Despite the fact that some of the data reported by Sachs et al (**7,8**) may be open to question, there appears to be enough evidence compiled by Muir et al and Grobe et al (**2,6**) in addition to the work reported by Sachs et al (**7,8**), to indicate that there probably does exist an inversion between elastic limit and hardness.

Further data that tend to corroborate this assumption are presented by Drutowski and Mikus (**9**), who investigated the effect of per cent retained austenite on rolling friction. Specimens of hardened 52100 steel were prepared with amounts of retained austenite from 0 to 18.4 per cent. The hardness of these specimens varied from Rockwell C 58 to C 64. Elastic-limit measurements taken on these groups of 52100 steel revealed a definite inversion between elastic limit and hardness. Drutowski and Mikus (**9**) found that, with retained austenite levels up to 3.9 per cent, the elastic limit remained at maximum value. However, when the structure contained 7.4 per cent retained austenite, the elastic limit fell off drastically. This occurred despite the fact that the specimens with 7.4 per cent retained austenite had a Rockwell hardness of C 62, while the specimens with 0-per cent retained austenite had a Rockwell hardness of only C 58. Evidently, differences in strain rate, stress pattern, and perhaps other factors imposed on the test specimen in determining mechanical properties such as elastic limit, as compared to hardness, are sufficient to produce an inversion of the material properties. It is also apparent that the rolling-fatigue process bears a close enough relation to the hardness measuring process that a correlation does exist between rolling fatigue life and hardness. It follows from this that a similar correlation between mechanical properties such as elastic limit and fatigue life cannot exist.

Deformation and Wear Studies:

Deformation traces of the test specimens were made on a Talyrond contour tracer. By use of this machine, the effect of the number of stress cycles and test conditions on permanent deformation could be studied. Deformation produced on surfaces in rolling contact takes three basic forms: (1) elastic deformation due to contact stresses; (2) plastic deformation due to excessive stresses and repeated loading, and (3) wear of the contacting surfaces due to relative sliding. Factors (2) and (3) result in the permanent alteration of the ball that can be measured after a test. Figure 7(*a*) is a diagram of the transverse section of a ball surface showing this condition. A diagram of the Talyrond trace of this transverse section which magnifies deviations from a true sphere would appear as shown in Fig. 7(*b*). The profiles in Figs. 7(*a*) and (*b*) show that the original surface contour has been raised immediately adjacent to the running track. The region of increased volume extends approximately one track width on either side. If wear did not take place, the volume of material added to the region adjacent to the track would equal the volume of material displaced from the track itself, since steels are essentially incompressible. However, this is not the case. The volume lost in Fig. 7 is much greater than the volume gained. The difference represents material removed by wear.


Profile
Sphere
Chord
(a)
Deformation
Deformation, Plus Wear
Profile, Deviation Magnified x2000
True Sphere, Magnified x30
Cord, Deviation from Sphere, Magnified x2000
(b)

(*a*) Schematic cross-section, sphere and deviation at same magnification.
(*b*) Talyrond profile trace, deviation highly magnified.

FIG. 7.—Cross-Section of a Postrun Ball Specimen Track (not to scale).

Radial distances on a Talyrond trace, such as those shown schematically in Fig. 7(*b*), are magnified about ×2000, while circumferential differences are magnified about ×30. From Fig. 7(*b*) it can be seen that due to the convergence of the transverse distance and areas close to the center, the areas of deformation

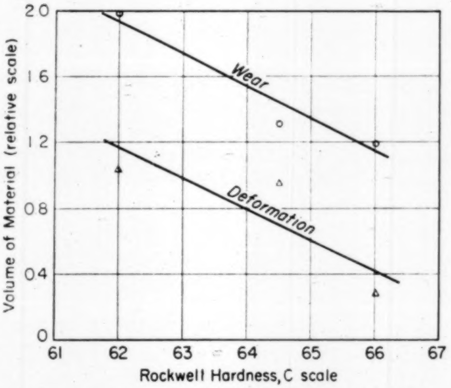


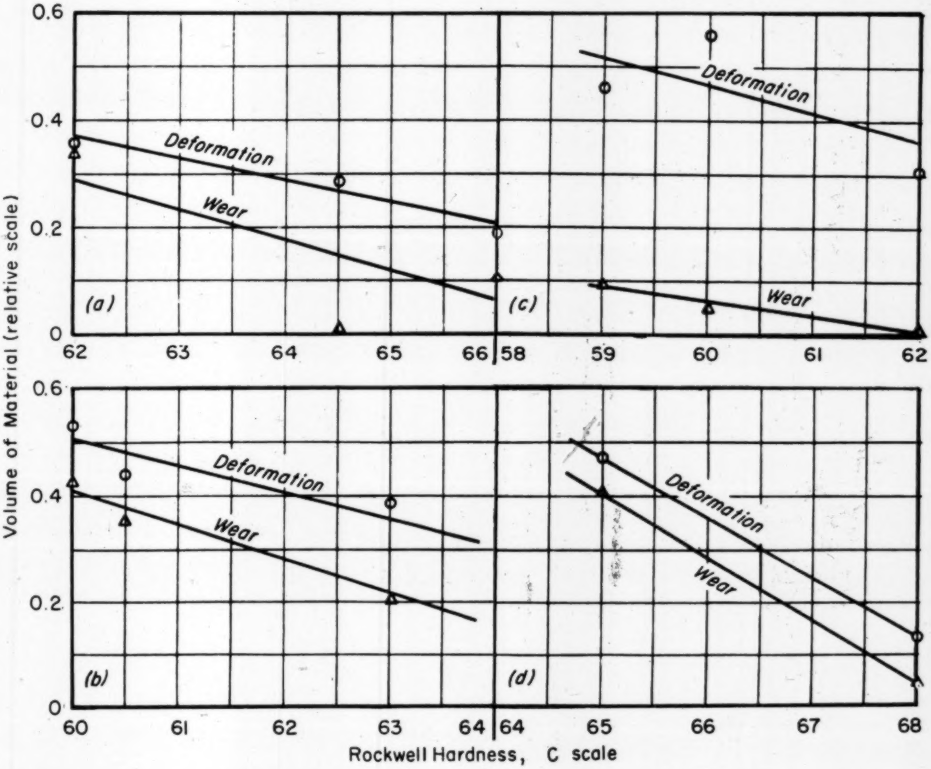
FIG. 8.—Deformation and Wear of AISI M-1 Tool-Steel Ball Running Tracks as Functions of Hardness After 1.7 × 10^9 Stress Cycles at 800,000-psi Maximum Hertz Stress and 0-deg Contact Angle.

and wear cannot be measured directly. To measure these areas accurately, the Talyrond trace was projected at ×5 and drawn on polar grid paper. The converging transverse distances were then adjusted mathematically to compensate for the unequal magnification. The areas of deformation and of deformation plus wear on the trace were measured by use of a planimeter and the volumes were calculated. The wear volume is the differences between these two volumes.

Deformation and wear data obtained using M-1 balls with Rockwell hardnesses C 62, C 64.5, and C 66 run in the fatigue spin rig are shown in Fig. 8. These specimens were run for about 1.7 × 10^9

(a) AISI M-1

(c) Halmo



(b) AISI M-50

(d) WB-49

FIG. 9.—Deformation and Wear of Tool-Steel Ball Running Tracks as Functions of Hardness After 10,000 Stress Cycles at 750,000-psi Maximum Hertz Stress and 40-deg Contact Angle.

stress cycles under the same test conditions as the M-1 fatigue specimens reported in Fig. 2. A general trend toward decreased wear and deformation volume with higher material hardness is observed. Balls of the three hardness levels of M-1, M-50, and Halmo steels and the two highest hardness levels of WB-49 steel were run in the five-ball fatigue tester to obtain additional deformation and wear data. These balls were run at a 40-deg contact angle for a total of 10,000 stress cycles at 750,000-psi maximum Hertz stress. The data obtained with these balls are shown in Fig. 9. For each of the four materials, a general trend toward decreased deformation and wear is observed with increasing hardness.

Discussion of Deformation and Wear Results:

The data of Figs. 8 and 9 indicate a definite correlation between hardness and resistance to plastic flow. For each of the four alloys studied, the hardest material offered the greatest resistance to plastic deformation. Hardness seems to be the important property here, and in this respect these results are in agreement with those of Drutowski and Mikus (**9**), who found that the extent of plastic deformation in SAE 52100 plates correlated with hardness at maximum Hertz stress levels above 600,000-psi, and not with elastic limit. This seems to point to the fact that hardness is the important property at the extreme stress levels of rolling-contact fatigue tests, whereas at lower stress levels, elastic limit may be the important property. One would certainly expect beforehand that plastic deformation could be related to elastic limit, since elastic limit is a measure of the susceptibility of a material to plastic deformation. However, the experimental data on rolling-contact fatigue life do not indicate that this is the case. And the breakdown in the expected relation between fatigue life and such mechanical properties as elastic limit probably comes about because of the extreme stress levels at which rolling-contact specimens are tested.

Metallographic Structure:

The different tempering temperatures used to produce the variation in hardness for each of the four materials studied produced only minor modifications in metallographic structure. The softer balls tend to have larger, more numerous precipitated carbides and greater definition of tempered martensite. Previous metallographic studies such as that of Bear and Butler (**10**) have shown that carbides acting as stress raisers can cause severe localized matrix damage that produces incipient fatigue cracking. Thus, variation in carbide size and distribution caused by variations in tempering temperature may be an important contributor to the observed variations in fatigue life.

Table II contains data on the per cent by volume of retained austenite for the materials used in this investigation. The per cent retained austenite increased with decreasing tempering temperature and increasing hardness. One of the practical requirements of the bearing material heat-treatment schedule is control of retained austenite. Volume changes resulting from in-service austenite transformation produce dimensional instability which is detrimental to precision bearing operation. Drutowsky and Mikus (**9**) have shown that extremely high percentages of retained austenite in SAE 52100 steel produce drastic reductions in elastic limit. Thus, aside from the dimensional instability problem, the percentage of retained austenite in the microstructure may influence rolling-contact fatigue life of tool steels. Since both hardness and the amount of retained austenite are controlled by tempering temperature and successive heat treatments, it is difficult to evaluate independently the effect of retained austenite on fatigue life. However, in the experiments reported herein, if there was any detrimental effect on fatigue life because of the presence of greater amounts of the austenite phase in the microstructure of the harder specimen groups, it was of lesser magnitude than the beneficial effect of the higher material hardness.

Each of the four materials used in this investigation were specimens fabricated from very clean vacuum-melted heats of material. Cleanliness ratings for the four steels studied are given in Table IV. Because of possible differences in behavior between clean and dirty steels, the conclusions reached apply only to very clean vacuum-melted heats. It is possible that relatively dirty steels will show a deterioration in fatigue performance as hardness increases above a certain value. The effect on fatigue of inhomogeneities such as inclusions may be magnified with the reduced ductility obtained at high hardness levels.

TABLE IV.—NONMETALLIC INCLUSION RATINGS OF TEST MATERIALS.
(Jernkontoret charts (ASTM Recommended Practice E 45 – 51)[a])

Material	Thin series[b]				Thick series[b]			
	A	*B*	*C*	*D*	*A*	*B*	*C*	*D*
AISI M-1	0	1.0	0	1.5	0	0	0	0
AISI M-50	0.5	1.5	0.5	2.0	0	0.5	0	0.5
Halmo	0.5	2.0	0.5	2.0	0	0	0	0.5
WB-49	1.5	0	0	1.0	...	...	...	...

[a] Recommended Practice for Determining the Inclusion Content of Steel (E 45 – 51), 1958 Book of ASTM Standards, Part 3, p. 529.

[b] Designation: A = sulfides, B = aluminates, C = silicates, D = oxides.

Statistical Significance of Results:

The validity of experimental results which are analyzed statistically can only be determined by estimating the confidence which can be placed in the results. There are many methods available for calculating confidence levels for fatigue data. Johnson (11) gives a thorough treatment of this problem, and his methods have been used to examine these data. The basic question to be answered in comparing the fatigue data obtained with two materials or, in this case, with two different hardnesses of the same material, is how confident can we be that one material is really superior to the other?

For the four materials investigated, comparing the hardest group with the softest, the following confidence levels for the 10 per cent life were calculated:

M-1	93.5 per cent
M-50	81.5 per cent
Halmo	91.5 per cent
WB-49	95.5 per cent

This indicates that, for each of the four materials, the chances that the hardest group is superior to the softest group exceed 80 out of 100. The confidence levels associated with the hardest and the second-hardest group of specimens for each material are lower because of the smaller differences in fatigue lives. However, it is significant to note that the ten per cent lives for all four materials increased continuously with hardness without any exceptions. In order to achieve high confidence levels for fatigue data not exhibiting large differences in lives, it is necessary to test a very large number of specimens. In many instances this is not economically feasible.

SUMMARY OF RESULTS

Two bench-type rolling-contact fatigue testers, the fatigue spin rig and the five-ball fatigue tester, were used to determine the rolling-contact fatigue life at room temperature of groups of AISI M-1, AISI M-50, Halmo, and WB-49 alloy steel balls tempered to various hardness levels. Tests were run at a maximum Hertz stress of 800,000 psi using a synthetic diester lubricant that met the MIL-L-7808 specification. Material hardness, resistance to plastic deformation of specimens in rolling contact, and previously published tensile and compressive strength data for bar specimens of the same heats of material were compared with the fatigue-life results. The following results were obtained:

1. The 10 per cent rolling-contact fatigue life, and thus the load-carrying capacity for each of the four alloy compositions studied, increased continually with increasing hardness. The improvement in load capacity was on the order of 30 to 100 per cent over the hardness range tested.

2. These fatigue results did not correlate with previously published tensile and compressive strength results for the bar specimens, which showed an apparent maximum at intermediate hardness values.

3. Resistance to permanent plastic deformation of specimens in rolling contact showed a general increase with increasing hardness. Since the stress pattern in the hardness test is similar to that of bodies in rolling contact, some measure of resistance to plastic deformation may be a qualitative estimate of relative fatigue lives of different heat treatments of a given alloy composition.

4. Only minor differences in metallographic structure were observed for the various hardness levels of each alloy considered. The softer balls tended to have more and larger fine precipitated carbides and greater definition of tempered martensite. Per cent retained austenite increased with higher hardness.

REFERENCES

(1) E. G. Jackson, "Rolling Contact Fatigue Evaluation of Bearing Materials and Lubricants," *Transactions,* Am. Soc. Lubrication Engrs., Vol. 2, No. 1, p. 121 (1959).

(2) Hugh Muir, B. L. Averback, and Morris Cohen, "The Elastic Limit and Yield Behavior of Hardened Steels," *Transactions,* Am. Soc. for Metals, Vol. 47, pp. 380–399 (1955); Discussion, pp. 399–407.

(3) T. L. Carter, E. V. Zaretsky, and W. J. Anderson, "Effect of Hardness and Other Mechanical Properties on Rolling Contact Fatigue Life of Four High Temperature Bearing Steels," *NASA TN D-270,* Nat. Aeronautics and Space Administration, (1960).

(4) E. F. Macks, "The Fatigue Spin Rig—A New Apparatus for Rapidly Evaluating Materials and Lubricants for Rolling Contact Bearings," *Lubrication Engineering,* Vol. 9, No. 5, Oct. 1953, pp. 254–258.

(5) R. A. Baughman, "Effect of Hardness, Surface Finish, and Grain Size on Rolling Contact Fatigue Life of M-50 Bearing Steel," *Paper No. 59-LUB-11,* Am. Soc. Mechanical Engrs., (1959).

(6) Arthur H. Grobe and George A. Roberts, "The Bend Test for Hardened High Speed Steel, *Transactions,* Am. Soc. for Metals, Vol. 40, pp. 435–471 (1948).

(7) G. Sachs, R. Sell, and V. Weiss, "Tension,

Compression, and Fatigue Properties of Several SAE 52100 and Tool Steels Used for Ball Bearings," *NASA TN D-239*, Nat. Aeronautics and Space Administration (1960).

(8) G. Sachs, R. Sell, and W. F. Brown, Jr., "Tension, Compression, and Fatigue Properties of Several Steels for Aircraft Bearing Applications," *Proceedings*, Am. Soc. Testing Mats., p. 635 (1959).

(9) R. C. Drutowski and E. B. Mikus, "The Effect of Ball Bearing Steel Structure on Rolling Friction and Contact Plastic Deformation," *Paper No. 59-LUB-2*, Am. Soc. for Metals (1959).

(10) H. Robert Bear and Robert H. Butler, "Preliminary Metallographic Studies of Ball Fatigue Under Rolling Contact Conditions," *NACA TN 3925*, Nat. Advisory Committee for Aeronautics (1957).

(11) Leonard G. Johnson, "The Statistical Treatment of Fatigue Experiments," General Motors Research Laboratories, *GMR-202*, April, 1959.

DISCUSSION

MR. HARRY R. NEIFERT.[1]—The single-element tests presented by the author are very similar to those published by other investigators in that the stress levels at which these single elements are tested are extremely high compared with the actual stresses that exist in rolling-element bearings. This raises a question as to the correlation between single-element data and the life characteristics of the complete bearing. For example, from the Talyrond trace shown in the paper, we know that considerable plastic deformation existed so that after, say, 1 cycle of revolution the stress to which the element was initially subjected was then immediately changed. The stresses we are talking about as acting on the single element during the test did not actually exist for the duration of the test run.

I should like to ask the author whether he has tried to correlate any of these data with what he would get on full-complement bearings.

Secondly, on the basis of the author's tests, would he be willing to say that bearings made from one of these steels at a particular hardness level would give him optimum operating characteristics?

MR. W. J. ANDERSON (*author*).—First of all, I agree with Mr. Neifert that these stress levels are extremely high. We gave considerable thought to the stress level at which we should conduct tests when we started fatigue work several years ago.

In the beginning we examined the type of failure we got and the resulting scatter of the fatigue data which is given by the slope of the Weibull plot. The type of failure we got agreed, or compared very closely, with those obtained in full-scale bearings as to characteristics of the spall, etc. Also, the scatter in the data approximated that for normal bearing tests.

We also tested at a number of stress levels, and the stress-life relationship obtained from the fatigue spin rig was on the order of the inverse ninth to tenth power, which compares very favorably with ball bearings. On this basis we assumed that the data we were getting could be extrapolated down to much lower stress levels and still be valid.

Just recently we completed fatigue tests in the fatigue spin rig on nine different lubricants. Full-scale bearing tests were run with five of the nine lubricants at the New York Naval Ship Yard, sponsored jointly by the Office of Naval Research and Wright Aeronautical Development Division. We have correlated the fatigue life obtained in the spin rig with the results obtained in those bearings. The relative fatigue life, or load capacity, obtained in the spin rig agreed fairly well with the data obtained in the full-scale bearings.

---

[1] Chief Engineer, Physical Laboratories, Timken Roller Bearing Co., Canton, Ohio.

In regard to optimum life, we think these data are valid for reasonable heat treatment of material. This may not be true if you test a material which has been drastically quenched without tempering but all these materials were double-tempered for 2 hr, so that in none of these cases was the ultimate hardness obtained. For the hardness ranges in which we investigated fatigue life we believe that the best fatigue life will be obtained with the highest hardness.

This agrees somewhat with the existing full-scale bearing data. Data obtained by Marlin-Rockwell Corp. with SAE 52100 steel bearings (given in reference (3) of the paper) indicate that maximum life is obtained with maximum hardness.

MR. SAMUEL J. ROSENBERG.[2]—I wonder if some of these results could be explained in the light of the theoretical stresses noted. These stresses were calculated in accordance with the Hertz formulae, which are based entirely upon the assumption that any deformation occurs strictly in accordance with Young's modulus. This means that any material that has a lower elastic limit will, if the stresses are projected in accordance with Hertz formulae, show stresses that are significantly different from the stresses that actually exist, as compared to a material that has a higher elastic limit, for the same conditions of loading. When materials of different hardnesses are compared, this is a factor that must have some effect. Even though the loads and, therefore, the theoretical calculated stresses are the same, the materials may actually contain entirely different stresses during the test itself because of plastic deformation of the softer material.

MR. ANDERSON.—I quite agree that the actual stresses that exist might be quite different from those derived from a simple Hertz calculation. The stress is affected not only by material properties but also by such conditions as surface tangential forces. Smith and Liu (12) have studied this for a two-dimensional case, and they have found that the location of the maximum shearing stress moves toward the surface as the surface tangential force increases. If the friction coefficient is 0.09 or greater, the maximum shearing stress might very well be at the surface rather than below the surface. This is one factor, and material properties are another.

I am not so sure that the elastic limit or any other property is the property of significance. For instance, the assumption that a material with a low elastic limit will deform plastically to a greater extent than one with a high elastic limit leads to the conclusion that the low elastic limit material will be subjected to lower stresses. It follows that the material with low elastic limit should have better fatigue life, other things being equal, but this may or may not be true. Recent work by Drutowski and Mikus (13) has indicated that at low stress levels elastic limit seems to control the amount of plastic deformation that takes place and, at higher stress levels, hardness becomes more important and elastic limit becomes less important.

MESSRS. G. J. MOYAR[3] AND JODEAN MORROW[3] (*presented in written form*).—Over the past three years, a study of the failure of bodies in rolling contact has been made at the University of Illinois, including an attempt to relate pitting failure to ordinary fatigue failure of materials. The contact fatigue behavior of 52100 steel was related to the torsional fatigue properties for the same steel and hardness condition (14). This was done by considering the criterion for fatigue failure in rolling contact to be the maximum alternating shear stress. Other factors, such as size effect and state of stress, were also taken into account. This analysis has been used to predict the life of full-scale M-50 ball bearings (**15**) from tests of the same material made in the General Electric rolling-contact rig (**16**).

---

[2] Metallurgist, National Bureau of Standards, Washington, D. C.

[3] Research Associate and Associate Professor, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

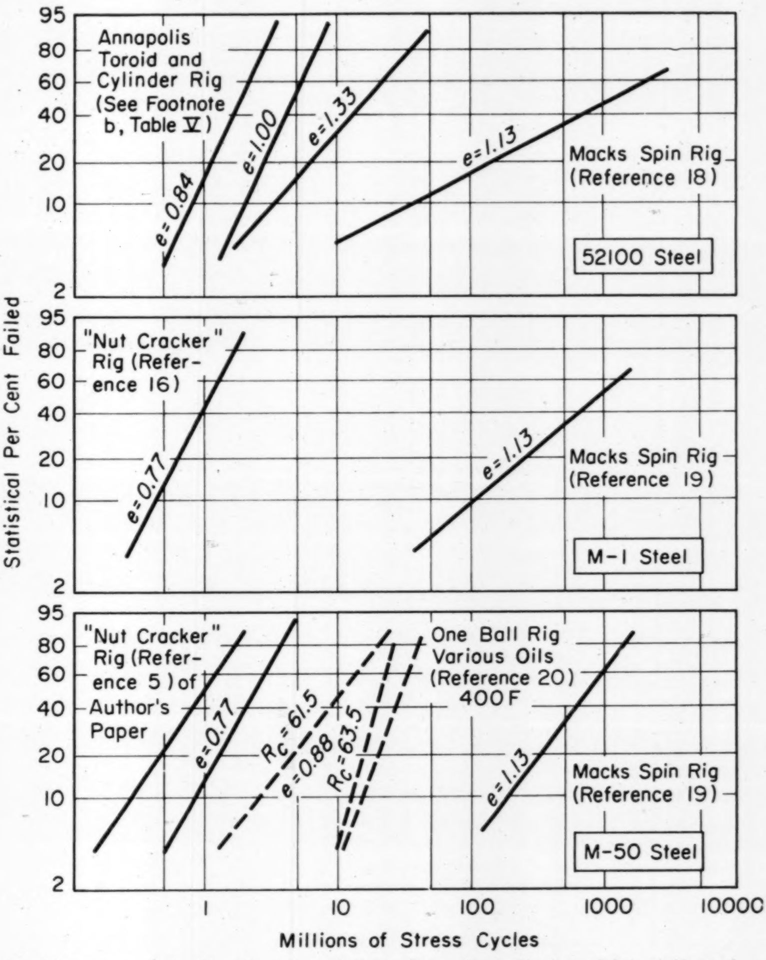


FIG. 10.—Summary of Rolling-Contact Fatigue Data from Various Rigs. Adjusted to σ_{max} = 800,000 psi.

For the purposes of economy and speed of evaluation, simulated bench rig testers of various designs have been employed in a number of investigations, particularly in the past ten years. The aim of such studies has been to relate the behavior of materials in these rigs to their behavior as actual rolling elements. Yet, a summary of some of these rig data reveals that the numerical correlation from one rig to another in terms of pitting endurance is, in itself, poor.

The authors' results and other data

TABLE V.—DETAILS OF TEST CONDITIONS.

Material Type	Rig Type	e^a	References	Material Modifications (Rockwell Hardness, C-Scale	Lubricant	Unadjusted Maximum Stress, psi	Ambient Temperature, deg Fahr	Stress Frequency, cpm	Number of Specimens Failed
52100	Toroid and cylinder	0.84	[b]	Rockwell hardness C 61 to 63	Navy designation 2190 T oil	840 000	90	1 780	26
52100	Toroid and cylinder	1.00	[b]	Rockwell hardness C 61 to 63	Navy designation 2190 T oil	851 500	90	1 780	13
52100	Toroid and cylinder	1.33	[b]	Rockwell hardness C 61 to 63	Navy designation 2190 T oil	826 000	90	1 780	21
52100	Macks spin rig	1.13	**(18)**	Commercial melt SAE grade 1 balls	SAE 10 min oil, 5 mm per hr	750 000	room temperature	163 000	74 balls, 27 fail
M-1	Nut-cracker	0.77	**(16)**	Commercial air melt, Rockwell hardness C 61	Mil-L-7808	725 000	room temperature	30 000	4
M-1	Macks spin rig	1.13	**(19)**	Induction vacuum, 2.8 per cent retained austenite, ASTM grain size 8, Rockwell hardness C 62	Mil-L-7808	800 000	room temperature	178 500	17
M-50	Nut-cracker	0.77	**(5)**	Commercial air melt, ASTM grain size 10.4, Rockwell hardness C 62	Mil-L-7808 Mil-L-7808	732 000 732 000	room temperature	30 000	4
M-50	Nut-cracker	0.77	**(5)**	Commercial air melt, ASTM grain size 3.4, Rockwell hardness C 62	Mil-L-7808	732 000	room temperature	30 000	4
M-50	Macks spin rig	1.13	**(19)**	Induction vacuum, ASTM grain size 8, Rockwell hardness C 62	Mil-L-7808	800 000	room temperature	178 500	12
M-50	1 ball rig	0.88	**(20)**	...	...	730 000	400	...	...

[a] e = ratio of contact ellipse axis in the rolling direction to the axis in the transverse direction.

[b] Personal communication with W. Smith and W. Greenert, U. S. Naval Engineering Experiment Station, Annapolis, Md.

obtained in the same rig are shown for three different materials in the accompanying Fig. 10. Also shown in this figure are results obtained from other bench rigs for nominally the same material. Table V lists the pertinent test conditions. Adjustment has been made to the same maximum Hertz stress according to the method referred to by the authors. The obvious conclusion from these figures is that the Macks spin rig gives lives that are longer than any of the other bench rigs shown.

The authors apparently mean by the word "correlation" that the B_{10} life for their rig will rank materials in the same relative order as the hardness test. In a stricter sense, the word "correlation" implies quantitative agreement between the results from the bench rig and tests of full-scale bearings. If only qualitative correlation is desired, Sachs (reference (8) of the paper) has shown that such correlation exists between "ordinary" fatigue tests of the bearing material and the full-scale bearings.

We should like to ask if the authors have ever attempted a quantitative correlation with their spin rig data and full-scale bearings. Furthermore, we ask if they have tested the same material under similar conditions in their five-ball rig and request that they include in their closure the results of these tests if they are available.

Some of the apparent discrepancies among the results of various rigs may be explained on the basis of configuration effects as treated in reference (14). In this analysis, rolling elements having a larger configuration index, e (ratio of contact ellipse axis in the rolling direction to the axis in the transverse direction) should have a longer life at the same maximum contact stress. However, the discrepancy is so large that other factors must be important. Among the possible sources of discrepancy are:

Load and Nominal Stress Determination.—In load determination, aerodynamic and frictional effects are ignored, and, as in all bench rigs, an elastostatic solution is used to compute the nominal maximum stress. Whatever deviation occurs from the ideal, including possible reduction of contact stress due to lubricant films, will be more pronounced in the spin rig because of the large velocities. Increase in the statistical variability (lower Weibull slopes) also attests to possible reduction in critical stress (17).

Determination of Stress Cycles.—The kinematics of the ball motion in the spin rig may be such that significant sliding occurs, and thus a point in the ball path experiences fewer stress cycles than pure rolling would indicate.

Frequency.—In general, the higher the frequency the longer the fatigue life of materials. This effect is usually considered to be small, but since the frequency of the spin rig is of the order 150,000 stress cpm it is conceivable that the increased frequency in the Macks spin rig allows the material to last longer without fatigue failure.

We should like to ask if a systematic investigation of the importance of factors such as those listed above has been made. If so, can some idea be given of the magnitudes of the effects? The rig as originally designed had provision for friction torque determination. Have such measurements been made?

About 50 years ago various hardness testers began to appear in the field of materials testing. An effort was made to devise a short-cut method to determine the strength of a material. It seems that we are passing through a similar phase in connection with contact fatigue. Various bench rigs analogous to the various hardness testing machines are being made, and numerous tests are being run to evaluate materials for use in rolling elements. If something approaching a fundamental property of materials is ever to be established which is related to the resistance to rolling contact fatigue, critical evaluation of these various bench rigs must be made.

In closing, the writers should like to make an appeal to investigators working in this field to give some attention to standardization of such a test. We feel that it is particularly appropriate that this appeal should appear in a discussion to a paper in a society that is devoted to standardization.

MR. W. J. ANDERSON (*author's closure*).—The NASA work on rolling fatigue, which has been conducted wholly with bench-type fatigue rigs, has included the evaluation of materials and lubricants. It has not been the objective of this program to establish numerical ratings nor to attempt to extrapolate results to predict the life of full-scale bearings. Rather the results have been used only to indicate trends and, as Messrs. Moyar and Morrow state, to rank materials and lubricants.

If a reliable ranking of materials in rolling fatigue could be achieved by conventional fatigue tests such as those conducted by Sachs (reference (**8**) of the authors' paper), these means would certainly be employed; a mass of unpublished data accumulated by the bearing companies, as well as Sachs' data, however, indicate that this is not the case. Close examination of Sachs' rotating-beam fatigue tests reveals the following ranking for SAE 52100 heats:

1. Vacuum melt
2. Heat No. 2
3. Heat No. 3

On the other hand, Marlin Rockwell Corp. bearing data (reference (**8**) of the of the paper) obtained with ball bearings fabricated from the same heats of material reveals the following ranking:

1. Vacuum melt
2. Heat No. 3
3. Heat No. 2

The discussion of these results in Appendix II of reference (**17**) does not agree with the data. These data indicate that it is not possible to evaluate the rolling-contact fatigue properties of materials with conventional fatigue tests. In addition, the role of the lubricant in rolling-contact fatigue cannot be studied with conventional fatigue tests.

Initial test results obtained from the fatigue spin rig several years ago (**10,18**) revealed the discrepancy between the load capacity of the test ball obtained experimentally and that calculated from the Lundberg-Palmgren theory (**21**). This theory yields a load capacity of 687 lb for one ball of SAE 52100 material in the fatigue spin rig. The experimental data of reference (**10**) of the paper indicate a higher load capacity. This is the only meaningful method known to the authors for comparing rolling-contact fatigue data obtained in various bench rigs and in bearings.

An investigation of the various factors that could be responsible for the long fatigue life in the fatigue spin rig has failed to produce any positive answers. As stated by Messrs. Moyar and Morrow, the nearly circular contact area in the spin rig would lead to longer life. The almost pure rolling condition which exists in the spin rig, together with hydrodynamic effects produced by the extremely rapid cycling and high velocities, probably contribute to the long life. Unpublished data show that increasing the degree of slip or spin in the contact area reduces life, and that much greater than expected life is obtained with small ball bearings operated at extreme speeds (in excess of 100,000 rpm).

It is believed that no sliding of any significance occurs in the spin rig because of the sharply delineated rolling bands present on all but a few test balls (Fig. 3(*a*) of reference (**10**) of the paper). Accurate measurements of the track width indicate that the contact track is as predicted by Hertz theory. Calculation of centrifugal stresses in a test ball at 200,000 rpm (the approximate angular velocity of a spin rig ball about its own axis) reveals that these are nominal when compared to the Hertz stress. The alteration of the stress pattern by centrifugal stresses, however, may be a factor in prolonging life. Quantitative estimates of these effects have not been made. In most cases, it is not possible because of the dependence of ball load on speed in the spin rig.

Although the original paper on the spin rig (reference (**4**) of the paper) mentioned the measurement of rolling friction, it has not been possible to separate the angular deflection caused by aerodynamic drag from that due to the rolling resistance of the balls.

A word of caution is in order when attempting comparisons between rolling-contact fatigue data obtained with different heats of material. An order of magnitude difference in fatigue life can easily be obtained with two heats of the same material which otherwise appear to be identical by every known measurable property.

REFERENCES

(**12**) J. O. Smith and C. K. Liu, "Stresses Due to Tangential and Normal Loads on an Elastic Solid with Application to Some Contact Stress Problems," *Journal of Applied Mechanics*, Vol. 20, No. 2, p. 157, June, 1953.

(**13**) R. C. Drutowski and E. B. Mikus, "The Effect of Ball Bearing Steel Structure on Rolling Friction and Contact Plastic Deformation," *Journal of Basic Engineering*, Vol. 82, Series D, No. 2, p. 302, June, 1960.

(**14**) G. J. Moyar, "An Analysis of the Fatigue Strength of Surfaces in Rolling Contact," Department of Theoretical and Applied Mechanics, University of Illinois, June, 1958. Submitted in partial fulfillment of degree of Master of Science.

(**15**) J. Morrow, "Correlation of RC Rig Data with Full Scale Bearings," Consulting Report to General Electric Co., Evandale, Ohio, July, 1959.

(**16**) R. A. Baughman, "Experimental Laboratory Studies of Bearing Fatigue," *ASME Paper No. 58-A-235*, presented at the Annual Meeting, Am. Soc. Mechanical Engrs., Nov., 1958.

(**17**) G. M. Sinclair and T. J. Dolan, "Effect of a Stress Amplitude on Statistical Variability in Fatigue Life of 75S-T6 Aluminum Alloy," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 75, p. 867 (1953).

(**18**) R. H. Butler and T. L. Carter, "Stress-Life Relation of the Rolling-Contact Fatigue Spin Rig," *NACA Technical Note 3930*, Nat. Advisory Committee for Aeronautics, March, 1957, p. 18.

(**19**) E. V. Zaretsky and W. J. Anderson, "Relation Between Rolling Contact Fatigue Life and Mechanical Properties for Several Aircraft Bearing Steels," see p. 627; see also *NASA Technical Note D-270*, Nat. Aeronautics and Space Administration, March, 1960.

(**20**) E. G. Jackson, "Rolling Contact Fatigue Elevation of Bearing Materials and Lubricants," *Transactions*, Am. Soc. Lubricating Engrs., Vol. 2, No. 1, p. 121 (1959).

(**21**) G. Lundberg and A. Palmgren, "Dynamic Capacity of Rolling Bearings," *Acta Polytechnica* (M. E. Series), Vol. 1, No. 3 (1947).

LONG-TIME ATMOSPHERIC CORROSION TESTS ON LOW-ALLOY STEELS*

By H. R. Copson[1]

Synopsis

Atmospheric corrosion test data are given for a large group of low-alloy steels through 15.5 yr at a marine location at Kure Beach, N. C., through 17 yr at a marine location at Block Island, R. I., and through 18 yr at an industrial location at Bayonne, N. J. In the absence of alloying elements, corrosion was severe and early failure resulted. Alloying with copper, phosphorus, nickel, and chromium was beneficial. Such low-alloy steels quickly developed protective rust coatings, and the corrosion rates decreased rapidly to low values. During the last half of the test periods the corrosion rates of many of the better low-alloy steels ranged from 0.05 to 0.2 mil per yr, with the higher rates applying to the marine locations. The durability of bare, reasonably thick, boldly exposed specimens of good low-alloy steels is remarkable.

In May 1941 test specimens from a large group of low alloy steels were exposed to atmospheric corrosion at three locations. Two of these were marine locations, namely Kure Beach, N. C., and Block Island, R. I. At Kure Beach the test site was on low ground about 800 ft from the ocean. At Block Island the test site was on a bluff overlooking the open ocean and was about 15 miles from the mainland. The third site was an industrial location in the yard of a laboratory at Bayonne, N. J.

Data from the Kure Beach tests have been discussed (**1**)[2] but not reported in full hitherto. Data from the Block Island and Bayonne tests have been reported in detail through 9 yr of exposure (**2,3,4**). All the tests are now complete. The present paper summarizes the results through 15.5, 17, and 18 yr at the three locations, respectively.

The analyses and thicknesses of the steels are given in Table I. The steels are arranged in thirteen groups based on composition. Some of the materials were substantially free of alloying elements and in others the alloy content ranged upwards to 5 per cent. The main alloying elements were copper, phosphorus, nickel, and chromium, but some of the steels also contained molybdenum and higher than normal amounts of manganese and silicon. Thicknesses ranged from 0.028 to 0.083 in. Most but not all the steels were exposed at each of the locations.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Research Laboratory, International Nickel Co., Inc., Bayonne, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

TABLE I.—ANALYSES AND THICKNESSES OF STEELS.

Steel	Group	Description	Thickness in.[a]	Analysis, per cent									
				Carbon	Manganese	Silicon	Sulfur	Phosphorus	Nickel	Copper	Chromium	Molybdenum	Vanadium
No. 25	I	High-purity iron, plus copper	0.034	0.049	0.11	0.003	0.04	0.002	0.003	0.004	0.05	...	...
No. 9			0.069	0.022	0.035	0.002	0.02	0.006	0.006	0.007	0.001	...	...
No. 44			0.078	0.016	0.016	0.002	0.03	0.005	0.05	0.014	...	...	...
No. 45			0.071	0.020	0.020	0.003	0.03	0.006	0.05	0.020	...	...	...
No. 63			0.079	0.020	0.023	0.002	0.03	0.005	0.05	0.053	...	...	...
No. 54			0.049	0.02	0.07	0.01	0.03	0.003	0.18	0.10	...	...	...
No. 55			0.047	0.01	0.06	0.01	0.03	0.003	0.13	0.27	...	...	...
No. 10	II	Low-phosphorus steel, plus copper	0.050	0.041	0.22	0.002	0.04	0.010	0.05	0.02	0.01	...	...
No. 31			0.031	0.09	0.35	0.05	0.02	0.009	0.05	0.041	...	...	...
No. 5			0.029	0.043	0.39	0.007	0.02	0.007	0.01	0.05	0.07	...	...
No. 57			0.049	0.06	0.26	0.01	0.03	0.007	0.06	0.19	...	0.01	...
No. 6			0.030	0.046	0.40	0.005	0.02	0.007	0.000	0.21	0.07	...	...
No. 41			0.076	0.08	0.34	0.01	0.02	0.008	0.05	0.22	...	...	...
No. 43			0.080	0.06	0.32	0.01	0.02	0.010	0.05	0.24	...	...	...
No. 64			0.083	0.026	0.24	0.004	0.04	0.009	0.05	0.265	...	...	...
No. 40			0.064	0.03	0.17	0.01	0.03	0.006	0.05	0.27	...	...	...
No. 58			0.061	0.05	0.31	0.01	0.02	0.008	0.05	0.27	...	...	...
No. 42			0.076	0.08	0.42	0.01	0.02	0.010	0.05	0.30	...	...	...
No. 56			0.049	0.04	0.31	0.01	0.02	0.004	0.05	0.30	...	...	...
No. 7			0.028	0.038	0.40	0.004	0.02	0.006	0.004	0.50	0.07	...	...
No. 8			0.030	0.040	0.39	0.005	0.02	0.007	0.004	1.03	0.06	...	...
No. 1	III	High-phosphorus steel, plus copper	0.057	0.085	0.43	0.005	0.05	0.103	0.002	0.01	0.02	...	...
No. 26			0.034	0.021	0.05	0.18	0.03	0.105	0.002	0.01	...	...	...
No. 11			0.033	0.040	0.49	0.003	0.04	0.074	0.01	0.05	0.10	0.003	...
No. 2			0.063	0.095	0.41	0.007	0.06	0.106	0.003	0.20	0.03	...	...
No. 12			0.033	0.026	0.47	0.003	0.06	0.066	0.03	0.27	0.03	0.003	...
No. 16			0.034	0.09	0.43	0.005	0.03	0.058	0.24	0.36	0.06	...	...
No. 3			0.058	0.095	0.41	0.007	0.05	0.104	0.002	0.51	0.02	...	...
No. 4			0.062	0.095	0.40	0.005	0.06	0.107	0.001	0.94	0.02	...	...

(Continued on p. 652)

TABLE I.—(*Concluded*).

Steel	Group	Description	Thickness in.^{a}	Analysis, per cent									
				Carbon	Manganese	Silicon	Sulfur	Phosphorus	Nickel	Copper	Chromium	Molybdenum	Vanadium
No. 18......	IV	High-manganese and silicon steels, plus copper	0.032	0.33	0.78	0.31	0.04	0.043	0.03	0.03	0.06	...	...
No. 19......			0.035	0.16	1.32	0.24	0.03	0.018	0.03	0.03	0.12	...	0.093
No. 17......			0.029	0.25	1.35	0.18	0.03	0.022	0.01	0.28	0.03	...	...
No. 66......			0.077	0.17	0.67	0.23	0.03	0.012	0.05	0.29	0.14	...	...
No. 53......			0.056	0.10	0.69	0.26	0.04	0.072	0.06	0.43	...	...	...
No. 67......	V	Copper steel, plus chromium and silicon	0.062	0.07	1.52	0.75	0.02	0.017	0.08	0.10	0.40	...	...
No. 68......			0.060	0.22	1.13	0.65	0.02	0.06	0.04	0.12	0.50	...	...
No. 154......			0.049	0.17	0.79	0.89	...	0.017	0.10	0.22	0.60	...	...
No. 97......			0.038	0.13	0.66	0.79	0.02	0.029	0.11	0.22	0.56	...	...
No. 13......			0.035	0.072	0.27	0.83	0.02	0.140	0.03	0.46	1.19	...	...
No. 118......	VI	Copper steel, plus molybdenum	0.075	0.04	0.15	0.01	0.03	0.009	0.05	0.42	...	0.07	...
No. 120......			0.077	0.04	0.12	0.01	0.02	0.010	0.05	0.47	...	0.07	...
No. 38......			0.059	0.21	1.04	0.23	0.03	0.017	0.036	0.62	0.025	0.35	0.006
No. 121......			0.073	0.06	0.096	0.01	0.03	0.069	0.14	0.54	...	0.13	...
No. 48......			0.072	0.17	0.89	0.05	0.03	0.075	0.16	0.47	...	0.28	...
No. 32......	VII	Nickel steel	0.032	0.11	0.69	0.14	0.02	0.011	0.20	0.038	...	...	...
No. 27......			0.027	0.16	0.56	0.23	0.03	0.013	0.54	0.12	0.05	...	...
No. 33......			0.032	0.14	0.37	0.07	0.02	0.008	0.55	0.03	...	...	...
No. 34......			0.032	0.14	0.36	0.37	0.02	0.008	1.27	0.028	...	...	...
No. 28......			0.028	0.14	0.47	0.23	0.02	0.014	1.47	0.03	0.05	...	...
No. 147......			0.030	0.06	0.14	0.14	0.03	0.013	1.96	0.03	...	...	...
No. 46......			0.029	0.16	0.57	0.020	0.02	0.015	2.20	0.24	...	...	...
No. 35......			0.033	0.14	0.28	0.08	0.02	0.008	2.55	0.03	...	...	...
No. 20......			0.030	0.19	0.53	0.009	0.02	0.016	3.23	0.07	...	...	...
No. 62......			0.046	0.17	0.58	0.26	0.01	0.007	4.98	0.09	...	...	...
No. 36......			0.035	0.13	0.23	0.07	0.01	0.007	4.99	0.03	0.05	...	...
No. 30......	VIII	Nickel steel, plus chromium	0.030	0.13	0.45	0.23	0.03	0.017	1.18	0.04	0.65	0.01	...

No. 29......	IX	Nickel steel, plus molybdenum	0.030	0.16	0.53	0.25	0.01	0.013	1.84	0.03	0.09	0.24	...
No. 51......	X	Nickel steel, plus chromium and molybdenum	0.036	0.10	0.59	0.49	0.01	0.013	1.02	0.09	1.01	0.21	...
No. 52......			0.035	0.08	0.57	0.33	0.01	0.015	1.34	0.19	0.74	0.25	...
No. 37......	XI	Nickel-copper steel	0.063	0.09	0.66	0.044	0.02	0.106	0.64	1.12	...	...	...
No. 60......			0.042	0.12	0.57	0.17	0.02	0.01	1.00	1.05	...	...	...
No. 148......			0.030	0.05	0.17	0.13	0.03	0.014	1.05	0.89	...	...	...
No. 50......			0.028	0.09	0.48	1.00	0.03	0.055	1.14	1.06	...	...	...
No. 39......			0.067	0.11	0.43	0.18	0.02	0.012	1.52	1.09	...	...	...
No. 149......			0.031	0.05	0.32	0.20	0.07	0.043	1.58	0.86	...	...	...
No. 150......			0.032	0.05	0.13	0.13	0.03	0.012	1.77	0.93	...	...	...
No. 153......			0.029	0.06	0.27	0.20	0.01	0.013	2.78	1.83	...	...	...
No. 152......			0.032	0.06	0.14	0.41	0.03	0.015	2.82	1.30	...	...	...
No. 151......			0.032	0.08	0.20	0.21	0.01	0.015	3.12	1.14	...	...	...
No. 49......	XII	Nickel-copper steel, plus chromium	0.074	0.09	0.33	0.01	0.03	0.066	0.4	0.22	0.19	...	...
No. 16-1......			0.032	0.044	0.20	0.003	0.02	0.010	0.49	0.54	0.46	...	...
No. 59......			0.065	0.11	0.65	0.13	0.02	0.086	0.29	0.57	0.66	...	...
No. 47......			0.031	0.11	0.75	0.23	0.04	0.020	0.65	0.53	0.74	...	...
No. 14......			0.052	0.08	0.37	0.29	0.03	0.089	0.47	0.39	0.75	...	...
No. 119......	XIII	Nickel-copper steel, plus molybdenum	0.079	0.03	0.16	0.01	0.03	0.009	0.29	0.53	...	0.08	...
No. 65......			0.079	0.13	0.45	0.066	0.02	0.073	0.73	0.573	...	0.087	...

[a] Generally within ±0.002 in.

TABLE II.—WEIGHT LOSS, g per sq dm.

Steel	Kure Beach (800-ft site), N. C.					Block Island, R. I.						Bayonne, N. J.					
	0.5 yr	1.5 yr	3.5 yr	7.5 yr	15.5 yr	1.1 yr	3.3 yr	5.0 yr	7.2 yr	9.1 yr	17.1 yr	1.0 yr	3.0 yr	5.1 yr	7.1 yr	9.1 yr	18.1 yr
No. 25......	4.6	gone	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 9......	4.9	[b]	gone	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 44......	6.0	[b]	gone	...	...	67.0[b]	gone	...	...	...	...	16.6	57.9[a]	gone at 4 yr		...	...
No. 45......	3.5	8.2	[b]	gone at 5 yr		18.8	39.7[a]	...	...	...	...	8.1	14.1	19.7	[c]	31.8[a]	...
No. 63......	2.3	4.8	10.1	19.0	43.0	6.0	17.4	27.1	gone	...	...	5.8	10.1	11.3	...	16.1	21.7
No. 54......	2.5	5.0	9.7	16.4	29.8	5.5	14.7	22.5[a]	gone	...	...	5.1	8.3	10.3	12.8	13.5	...
No. 55......	2.4	4.8	8.9	15.0	25.6	4.3	10.8	20.8[a]	27.8[b]	gone	...	4.4	7.5	8.6	10.4	10.8	...
No. 10......	2.6	5.0	10.3[a]	19.7[b]	[b]	6.5[a]	18.2[a]	gone	...	...	...	5.6	9.3	11.3	14.5	17.7	...
No. 31......	2.2	4.4	8.8	15.2	[b]	4.4	10.8[a]	17.2[a]	gone	...	...	5.3	8.6	10.1[a]	12.7[a]	15.0[a]	...
No. 5......	2.6	4.6	9.6[a]	16.5[b]	gone	5.3	11.3[a]	gone	...	...	...	5.2	8.6	10.9[a]	14.0[a]	15.8[a]	...
No. 57......	2.3	4.4	8.4	14.4	23.4	4.0	9.2	14.2	19.7[a]	[b]	...	4.5	7.1	8.8	9.9	10.6	...
No. 6......	2.5	4.6	8.8	14.0	22.9[a]	4.7	9.9	12.5[a]	17.7[b]	...	...	4.2	6.7	8.2	9.6[a]	10.4[a]	...
No. 41......	2.1	3.9	8.2	13.9	23.4	3.7	...	13.7	19.3	23.4	...	4.1	...	8.0	...	10.3	14.8
No. 43......	2.2	4.0	8.1	13.7	23.1	3.8	...	[c]	17.2	22.2	...	4.1	...	8.0	...	10.5	14.7
No. 64......	2.2	4.0	7.9	13.6	22.1	3.9	...	13.5	...	21.5	...	3.9	...	7.6	...	10.2	14.3
No. 40......	2.1	4.2	8.2	13.8	23.6	3.9	...	16.6	21.4	25.8[a]	...	4.1	...	8.3	...	10.2	14.5
No. 58......	2.1	3.9	7.9	13.4	21.5	3.6	...	13.3	17.4	22.6	...	4.0	...	8.1	...	9.9	13.7
No. 42......	2.1	4.1	7.7	13.1	21.9	3.5	...	[c]	16.6	20.3	...	3.9	...	7.5	...	8.9	13.6
No. 56......	2.3	4.1	8.0	13.4	21.7	4.1	8.3	12.5	16.8[a]	21.5[a]	...	4.2	6.7	8.0	9.4	10.0	...
No. 7......	2.6	4.7	8.1	13.3	20.6[a]	4.1	8.7	12.1[a]	gone	...	...	4.0	6.2	6.9	8.3	9.4[a]	...
No. 8......	2.3	4.2	7.0	10.8	17.3[a]	...	...	...	...	...	...	...	...	...	...	...	...
No. 1......	3.1	9.8[a]	gone	...	...	40.8[a]	gone	...	...	...	...	13.5	32.0[a]	gone	...	...	...
No. 26......	2.1	5.4	10.5[a]	17.7[b]	gone	...	...	...	...	...	...	...	...	...	...	...	...
No. 11......	2.1	4.2	8.1	13.5	20.9[a]	3.7	9.3[a]	15.7[a]	gone	...	...	4.9	7.9	9.7	12.0[a]	12.6[a]	...
No. 2......	2.0	3.8	8.1	10.8	17.9	3.4	...	10.3	...	15.9	28.0[a]	3.8	...	7.0	...	8.7	12.0
No. 12......	2.0	3.9	7.4	12.0	19.0[a]	3.4	7.3	9.8	13.4[a]	16.5[a]	...	3.4	5.7	6.6	8.3	9.1	...
No. 16......	2.0	3.8	6.9	10.9	16.9[a]	3.2	7.2	9.5	12.1[a]	14.3[a]	...	3.4	5.9	6.9	7.4	8.3	...
No. 3......	2.0	3.8	6.6	10.3	16.5	3.2	...	8.9	...	14.2	21.8	3.4	...	5.7	...	7.2	10.3
No. 4......	2.3	3.7	5.9	9.0	13.5	...	...	...	...	...	...	...	...	...	...	...	...

No. 18	2.1	[c]	6.9	10.7	16.2[a]	3.3	7.2	10.9[a]	12.8[a]	16.0[a]	...	5.1	7.5	7.7	9.8	9.7[a]	...
No. 19	1.8	3.7	6.4	10.6	16.9[a]	2.8	6.4	8.8	11.1[a]	14.3[a]	...	5.2	7.6	8.9	11.1	11.3	...
No. 17	1.9	3.5	6.3	9.9	16.2[a]	3.0	6.3	8.3	10.9[a]	13.1[a]	...	3.9	5.7	6.3	[c]	8.3[a]	...
No. 66	1.9	3.4	6.6	10.6	16.6	3.0	...	9.0	...	13.9	24.3	4.1	...	7.5	...	10.1	12.5
No. 53	1.8	3.1	5.5	8.6	13.6	2.7	...	7.8	9.3	12.4	...	4.0	...	6.1	...	7.9	9.9
No. 67	...	...	...	...	...	2.5	...	5.5	...	7.5	11.9	4.1	...	6.2	...	7.9	...
No. 68	...	...	...	...	...	2.5	...	5.8	...	7.6	12.0	4.1	...	5.9	...	6.8	...
No. 154	...	...	...	...	...	2.3	...	4.8	...	6.4	9.9	3.0	...	4.1	...	4.3	...
No. 97	...	...	2.7	4.6	...	2.5	...	5.5	7.1	7.8	...	3.3	...	5.1	...	5.9	6.9
No. 13	1.6	2.6	3.4	4.6	6.3	2.4	...	4.8	5.7	6.5	...	1.8	...	2.6	3.2	3.5	4.1
No. 118	2.1	4.3	7.9	13.5	21.2	4.1	...	13.2	...	21.8	39.3[a]	4.0	...	7.7	...	9.4	13.4
No. 120	2.3	4.0	7.7	12.7	20.2	3.6	...	12.9	...	21.1	35.9[a]	3.9	...	7.2	...	8.8	12.6
No. 38	1.6	3.0	5.5	8.8	13.9	2.7	...	7.3	...	12.1	17.6	3.9	...	6.6	...	8.2	10.4
No. 121	1.5	3.5	6.1	9.9	14.6	2.9	...	8.7	...	14.0	21.6	3.0	...	5.9	...	7.1	9.6
No. 48	1.8	3.5	4.9	7.9	11.8	2.5	...	[c]	...	10.9	16.8	3.0	...	5.1	...	6.5	8.5
No. 32	2.1	3.9	7.4	13.1	21.5[a]	3.4	7.9	12.7[a]	17.2[a]	gone	...	4.7	7.2	8.8	10.5[a]	12.2[a]	...
No. 27	1.7	3.4	6.2	10.3	15.9[a]	2.9	6.6	9.5[a]	11.3[a]	14.9[a]	...	4.3	6.1	7.2	8.8	9.7	...
No. 33	1.9	4.1	7.7	12.6	20.4[a]	3.6	8.5	13.9[a]	gone	...	...	4.5	7.1	8.0	9.9	10.9[a]	...
No. 34	1.7	3.1	5.5	8.6	14.2[a]	2.6	5.2	8.3	9.9[a]	12.8[a]	...	3.8	5.7	6.6	7.7	8.1	...
No. 28	1.6	3.0	5.2	8.2	12.5[a]	2.4	5.2	6.8	8.3[a]	10.6[a]	...	3.3	4.6	5.8	6.9	7.9	...
No. 147	...	...	...	...	...	2.3	4.8	6.2	7.8	9.5[a]	...	2.7	4.5	5.2	6.6	7.2	...
No. 46	1.6	2.5	4.2	6.4	9.4	2.5	4.1	5.4	6.3	8.3	...	2.8	3.7	5.3	5.9	7.0	...
No. 35	1.7	2.9	5.1	8.0	12.2	2.5	5.0	6.6	8.5	10.2	...	3.3	5.1	6.2	7.0	[c]	...
No. 20	1.5	2.6	4.2	6.3	9.2	2.0	4.0	5.1	6.5	8.1	...	2.6	3.6	4.9	5.2	6.3	...
No. 62	1.2	2.0	3.0	4.2	6.1	1.7	...	3.9	...	[c]	7.9	2.3	...	3.2	...	4.9	5.6
No. 36	1.3	2.2	3.5	5.1	7.5	1.9	...	4.7	...	7.1	9.5	2.5	...	4.3	...	5.6	6.8
No. 30	1.6	2.6	4.5	6.8	10.5[a]	2.3	4.4	5.5	6.8	[a]	...	3.7	4.4	5.1	5.9	6.4	...
No. 29	1.4	2.8	4.3	6.5	9.8[a]	2.1	[c]	5.7	6.4	7.6	...	3.0	3.7	[c]	5.7	6.2	...
No. 51	1.5	2.1	3.2	4.5	6.5	2.0	...	4.4	4.7	5.4	...	2.4	...	3.2	3.9	3.8	4.3
No. 52	1.4	2.2	3.5	5.0	7.6	1.9	...	4.1	4.8	5.6	...	2.6	...	3.9	3.8	4.6	5.2

(Continued on p. 656)

TABLE 11.—(*Concluded*).

Steel	Kure Beach (800-ft/site) N. C.					Block Island, R. I.						Bayonne, N. J.					
	0.5 yr	1.5 yr	3.5 yr	7.5 yr	15.5 yr	1.1 yr	3.3 yr	5.0 yr	7.2 yr	9.1 yr	17.1 yr	1.0 yr	3.0 yr	5.1 yr	7.1 yr	9.1 yr	18.1 yr
No. 37	1.9	2.7	4.4	6.4	9.4	2.4	...	6.3	...	9.1	14.6	[c]	...	4.4	...	5.5	6.9
No. 60	1.7	3.0	4.7	7.2	10.6	2.6	5.2	7.3	...	10.1	...	3.3	4.5	5.3	6.1	6.7	...
No. 148	...	...	...	...	...	2.4	4.8	5.9	7.0	...	...	2.6	3.3	3.9	4.8	5.4	...
No. 50	1.6	2.9	3.3	4.3	5.6	2.2	3.8	...	5.4	6.3	...	2.3	2.9	3.3	3.9	4.8	...
No. 39	1.6	2.7	4.5	6.8	10.0	2.5	...	6.1	...	9.5	13.8	3.4	...	5.2	...	6.4	7.9
No. 149	...	...	...	...	...	2.3	...	5.9	6.6	7.6	...	2.5	...	3.5	4.9	5.3	8.1
No. 150	...	...	...	...	...	2.2	...	5.4	6.5	8.0	...	2.4	...	3.7	4.5	5.0	6.4
No. 153	...	...	...	...	...	2.0	...	4.5	5.3	6.6	...	2.1	...	2.9	3.2	[c]	4.2
No. 152	...	...	...	...	...	2.0	...	4.5	5.2	6.4	...	2.2	...	3.1	3.7	[c]	5.3
No. 151	...	...	...	...	...	2.0	...	4.6	5.6	6.4	...	2.0	...	3.0	3.4	[c]	4.8
No. 49	1.8	3.2	6.1	9.7	15.2	3.1	...	8.4	...	13.1	21.3	3.3	...	5.9	...	7.6	10.4
No. 16-1	2.0	3.8	6.9	10.9	15.9[a]	3.4	6.6	10.0[a]	10.6[a]	14.5[a]	...	3.7	5.1	5.9	7.0	7.3	...
No. 59	1.8	3.3	4.6	7.0	10.5	2.4	...	6.2	...	9.1	13.3	3.4	...	4.9	...	5.9	8.1
No. 47	1.7	2.6	4.2	6.4	9.3	2.4	4.2	5.5	6.4	8.5	...	3.4	4.0	[c]	5.0	5.8	...
No. 14	1.7	2.7	4.4	6.3	9.1	2.5	...	5.9	...	8.6	[c]	[c]	...	4.4	...	5.4	6.5
No. 119	2.0	3.8	7.0	11.5	18.2	3.4	...	10.8	...	17.0	29.4	3.6	...	6.7	...	8.0	10.9
No. 65	1.8	3.4	5.3	7.6	11.2	2.9	...	7.3	...	10.6	17.2	2.8	...	5.2	...	6.2	[c]

[a] Small perforations.
[b] Large holes.
[c] Attacked in cleaning.

Most of the specimens were 4 by 6 in. in size. They were identified by a two-hole numbering system, pickled, and weighed before exposure. They were mounted, using porcelain insulators, on metal racks similar to those adopted for use at ASTM test sites.[3] The specimens were inclined 30 deg from the horizontal. They faced south at all test locations. The direction at Block Island was such that the specimens faced the ocean while at Kure Beach the ocean was 800 ft east of the test racks in the general direction of the prevailing winds. The specimens were positioned in a random manner on the racks.

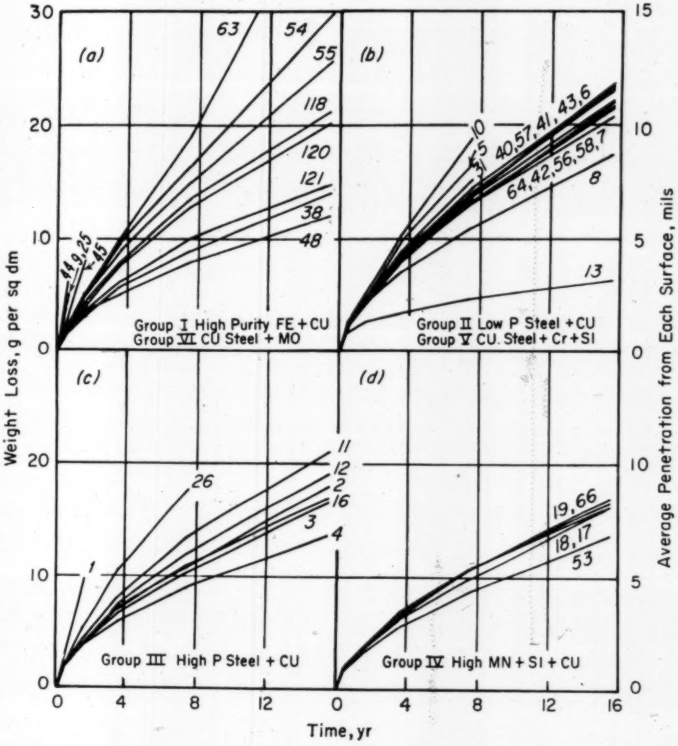


FIG. 1.—Weight Loss - Time Curves at Kure Beach (800-ft site), N. C., for Steels Substantially Free of Nickel.

RESULTS AT KURE BEACH

Sixteen specimens of each steel were exposed at Kure Beach. Two were removed at 0.5 yr, two at 1.5 yr, four at 3.5 yr, four at 7.5 yr, and four at 15.5 yr. The rust was removed by cathodic cleaning in hot inhibited sulfuric acid according to established procedures,[4] and the specimens weighed. Weight losses on replicate specimens were in excellent agreement. The average weight losses are given in Table II. The presence of any small perforations or holes is noted. The occurrence of perforations depended not only on composition but also on thickness. Weight losses are omitted where the specimens were starting to disintegrate, because these could be in substantial error. There was a wide variation in the performance of the materials. Some steels corroded away early in the tests, whereas others were in good condition at 15.5 yr.

---

[3] Report of Advisory Committee on Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 314 (1954).

[4] Tentative Method of Total Immersion Corrosion Test of Nonferrous Metals (B 185 – 43 T), Appendix I, 1958 Book of ASTM Standards, Part 3, p. 274.

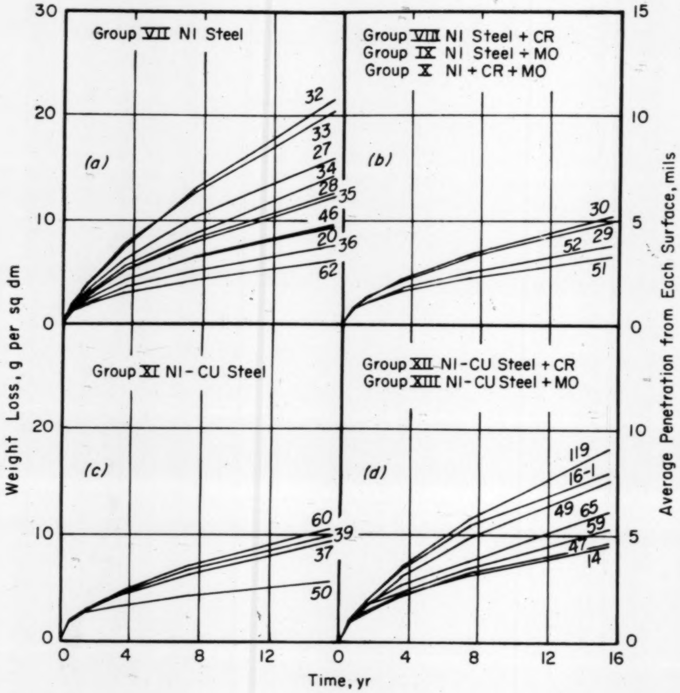


FIG. 2.—Weight Loss - Time Curves at Kure Beach (800-ft site), N. C., for Steels Containing Nickel.

Weight losses are plotted against time in Figs. 1 and 2. Figure 1 contains the data for steels which were substantially free of nickel, groups I through VI. Figure 2 contains the data for the remaining steels, all of which contained some nickel. The weight losses are given both as grams per square decimeter and as converted to the average penetration from each surface in mils. The smoothness of the curves attests to the consistency of the data.

Corrosion was rapid during the very early stages of the exposure tests and then began to decrease due to the formation of protective rust coatings (1,2). The protectiveness of the rust depended on the alloy content of the steel. With time the curves diverged and differences between the steels became marked. On the purer (unalloyed) materials, such as Nos. 9, 25, 44, and 45 of group I, the corrosion continued at a fast rate and early failure resulted. Some specimens of these materials with a thickness greater than $\frac{1}{16}$ in. had large holes or were disintegrating at 1.5 to 3.5 yr. With materials containing small amounts of copper, phosphorus, and other elements, such as Nos. 10, 31, 5, 1, and 26 of groups II and III, performance was better, but nevertheless the specimens were in poor condition long before the end of the tests. On the other hand, all specimens containing more than 0.2 per cent copper or nickel survived the test period. Some of these, which were lean in alloy content or which were quite thin initially, had perforations by 15.5 yr. With the more highly alloyed steels, however, corrosion rates had decreased to remarkably low values. Many of these still had about the same over-all thickness as at the start, with most of the corrosion occurring in small areas or pits. Pit depths were not measured on the Kure Beach specimens, but judging from measurements on specimens from the other marine location the deepest pits would run two to three times deeper than the average corrosion. The average corrosion on the two steels containing 5 per cent nickel, which were among the best steels in the test, was only 3.4 mils at 15.5 yr.

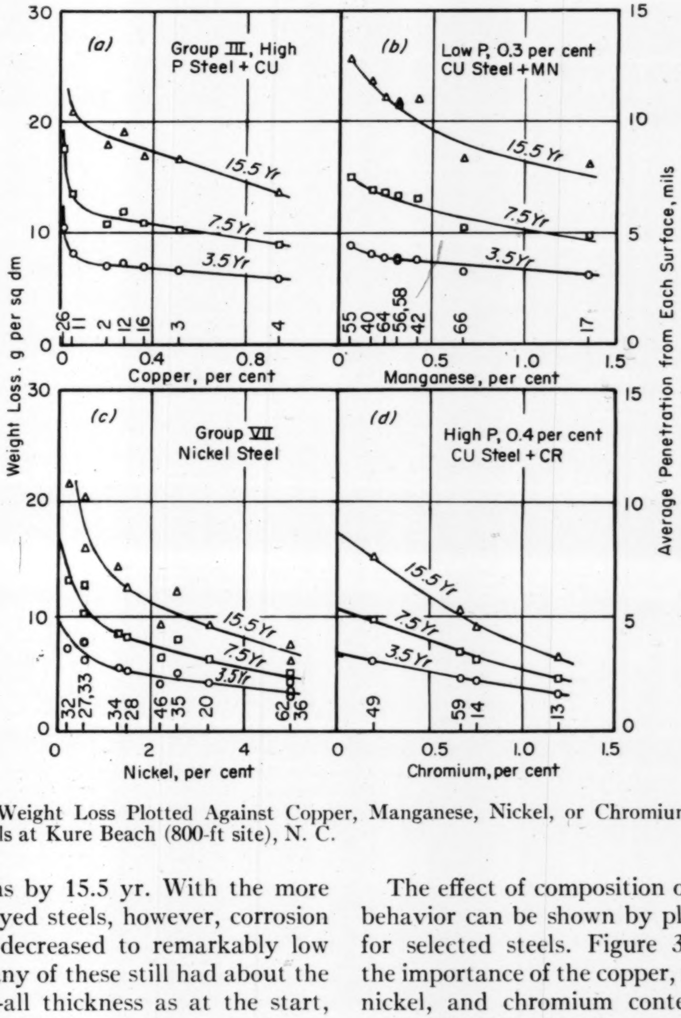


FIG. 3.—Weight Loss Plotted Against Copper, Manganese, Nickel, or Chromium Content of Certain Steels at Kure Beach (800-ft site), N. C.

The effect of composition on corrosion behavior can be shown by plotting data for selected steels. Figure 3 illustrates the importance of the copper, manganese, nickel, and chromium contents of the steels. The identification numbers of the steels used are indicated in the graphs. Data obtained at 3.5, 7.5, and 15.5 yr are plotted.

Figure 3(*a*) shows the effect of copper

TABLE III.—AVERAGE PIT DEPTH, mil[a]

Steel	Block Island, R. I.						Bayonne, N. J.					
	1.1 yr	3.3 yr	5.0 yr	7.2 yr	9.1 yr	17.1 yr	1.0 yr	3.0 yr	5.1 yr	7.1 yr	9.1 yr	18.1 yr
No. 44	...	...	...	...	...	...	17.5	...	...	...	...	...
No. 45	14.5	...	...	...	...	...	12.5	20.5	30.4	...	...	...
No. 63	11.7	19.5	31.9	...	...	...	9.0	14.0	18.6	...	19.5	24.5
No. 54	8.3	18.5	...	...	...	...	9.5	13.0	18.0	19.0	18.5	...
No. 55	6.7	13.0	...	...	...	...	7.5	11.5	15.5	17.5	15.0	...
No. 10	16.2[b]	...	...	...	...	...	9.0	13.5	19.7	20.6	22.0	...
No. 31	7.2	...	...	...	...	...	8.5	11.5	15.5[b]	...	...	...
No. 5	5.8	...	...	...	...	...	9.0	13.0	...	...	...	...
No. 57	6.2	13.5	15.5	24.5[b]	...	...	8.0	11.0	15.2	15.0	16.5	...
No. 6	6.2	11.0	...	...	...	...	7.0	9.5	12.9	15.3[b]	...	...
No. 41	7.0	...	19.9	24.5	30.6	...	8.0	...	13.6	...	14.9	12.5
No. 43	7.2	...	[c]	21.9	27.6	...	7.5	...	14.1	...	14.6	16.5
No. 64	7.0	...	18.9	...	31.3	...	7.5	...	12.7	...	14.0	16.7
No. 40	7.2	...	20.4	23.9	31.5[b]	...	8.0	...	12.6	...	15.1	16.6
No. 58	7.0	...	20.8	21.6	27.1	...	7.0	...	14.7	...	14.6	16.0
No. 42	7.5	...	18.5	24.5	25.0	...	6.5	...	12.7	...	14.8	14.6
No. 56	7.5	12.0	18.5	...	...	...	7.5	11.5	13.7	14.7	14.9	...
No. 7	5.3	11.0	...	...	...	...	7.0	10.0	12.8	13.8	...	...
No. 1	...	...	...	...	...	...	16.5	...	...	...	...	...
No. 11	6.5	...	...	...	...	...	9.0	13.0	15.1	17.3[b]	...	...
No. 2	8.0	...	15.1	...	22.6	...	6.5	...	12.0	...	14.0	13.0
No. 12	6.3	10.0	16.2	...	...	...	6.5	8.5	13.3	14.2	13.1	...
No. 16	5.8	10.0	12.3	16.5[b]	...	...	5.5	10.0	12.3	11.6	11.1	...
No. 3	7.6	...	12.3	...	20.6	27.9	7.5	...	11.5	...	12.1	14.2
No. 18	7.0	12.0	...	...	...	...	9.5	11.5	13.4	13.9	16[b]	...
No. 19	6.5	11.0	17.3	...	...	...	10.0	11.0	14.7	14.3	14.3	...
No. 17	6.0	10.0	13.2	...	...	...	8.0	9.0	13.3	13.4	14.5[b]	...
No. 66	6.2	...	13.7	...	20.1	30.9	8.0	...	12.3	...	14.1	16.9
No. 53	6.0	...	16.7	16.4	19.3	...	7.0	...	11.2	...	11.9	14.4
No. 67	4.3	...	11.4	...	12.3	15.3	7.5	...	13.5	...	13.6	...
No. 68	5.7	...	11.1	...	14.4	17.2	9.5	...	12.7	...	11.4	...
No. 154	6.8	...	9.7	...	11.8	16.2	7.0	...	11.0	...	9.4	...
No. 97	4.3	...	11.9	13.2	15.0	...	9.0	...	10.7	...	12.8	11.7
No. 13	4.7	...	11.5	11.6	14.3	...	6.0	...	10.5	9.9	10.4	13.0
No. 118	7.2	...	16.3	...	32.3	...	7.0	...	13.6	...	13.6	15.7
No. 120	7.8	...	18.0	...	35.0	...	8.0	...	11.9	...	14.6	17.4
No. 38	6.0	...	13.5	...	19.8	24.1	9.0	...	11.7	...	12.6	12.2
No. 121	6.2	...	14.8	...	20.9	24.9	6.5	...	9.6	...	11.6	16.0
No. 48	5.7	...	[c]	...	15.9	20.1	8.5	...	10.8	...	11.3	12.4
No. 32	6.0	13.0	...	...	...	...	8.0	11.0	13.2	14.3[b]	15.5[b]	...
No. 27	5.7	10.5	...	...	...	...	8.0	9.5	11.3	11.7	14.1	...
No. 33	6.2	13.0	...	...	...	...	9.0	11.0	14.7	14.7	15.3[b]	...
No. 34	6.6	10.0	15.9	17.0[b]	...	...	8.0	9.5	12.2	13.1	13.0	...
No. 28	4.7	9.0	13.3	14.0[b]	...	...	9.0	8.5	11.2	12.0	10.4	...
No. 147	5.8	8.5	11.2	13.5	15.0[b]	...	7.5	8.0	10.8	11.3	10.5	...
No. 46	5.0	8.5	10.1	13.8	13.1	...	7.0	7.5	10.4	9.9	10.1	...
No. 35	5.3	6.0	9.7	12.3	13.6	...	9.5	8.0	9.8	10.2	[c]	...
No. 20	4.7	5.5	8.5	11.8	13.3	...	8.0	6.5	9.2	9.8	9.0	...
No. 62	3.7	...	6.5	...	[c]	11.8	5.0	...	8.9	...	7.3	9.7
No. 36	5.0	...	5.2	...	10.8	12.9	5.5	...	7.8	...	8.1	11.4
No. 30	4.3	8.5	11.4	14.8	...	...	10.0	9.0	12.7	11.9	11.4	...
No. 29	5.0	[c]	8.9	14.2	17.3	...	7.0	8.0	[c]	8.9	10.6	...

TABLE III.—(*Concluded*).

Steel	Block Island, R. I.						Bayonne, N. J.					
	1.1 yr	3.3 yr	5.0 yr	7.2 yr	9.1 yr	17.1 yr	1.0 yr	3.0 yr	5.1 yr	7.1 yr	9.1 yr	18.1 yr
No. 51	5.0	...	9.3	10.1	13.4	...	7.5	...	10.8	9.0	10.6	10.8
No. 52	4.2	...	8.4	10.9	14.0	...	7.0	...	9.3	10.3	9.6	11.5
No. 37	4.8	...	12.0	...	15.0	18.4	6.0	...	9.7	...	10.3	10.8
No. 60	5.3	9.0	10.8	...	16.0	...	8.0	9.0	11.5	11.0	10.8	...
No. 148	4.5	8.0	9.7	14.7	...	...	7.0	6.5	10.8	9.8	11.0	...
No. 50	4.3	6.5	...	9.8	11.5	...	5.0	8.0	8.3	9.3	10.4	...
No. 39	7.0	...	10.6	...	14.5	22.1	8.0	...	9.8	...	10.8	12.1
No. 149	7.0	...	11.2	13.1	15.6	...	4.5	...	8.1	8.2	9.9	13.5
No. 150	5.0	...	9.9	13.3	14.0	...	7.0	...	9.9	9.2	9.6	12.4
No. 153	3.7	...	10.3	10.7	11.6	...	8.5	...	8.3	8.6	10.0	12.9
No. 152	5.0	...	10.8	10.4	12.0	...	7.0	...	8.9	9.4	8.6	10.6
No. 151	5.2	...	8.3	10.3	12.9	...	5.5	...	8.0	8.9	10.9	11.4
No. 49	7.2	...	12.4	...	17.8	27.0	8.0	...	11.1	...	11.6	12.6
No. 16-1	6.5	11.0	16.0[b]	...	...	...	9.0	8.0	11.8	11.9	12.3	...
No. 59	6.0	...	10.8	...	18.1	19.9	9.5	...	12.1	...	11.3	14.3
No. 47	6.7	9.5	11.2	12.9	15.5	...	8.0	9.5	[c]	10.4	11.6	...
No. 14	6.0	...	11.4	...	17.0	...	[c]	...	10.4	...	12.3	12.4
No. 119	5.8	...	14.7	...	28.1	35.4	6.0	...	12.2	...	13.5	15.0
No. 65	6.0	...	11.0	...	17.0	19.6	6.5	...	9.3	...	10.8	[c]

[a] Average of 4 deepest pits on skyward surface and 4 deepest pits on groundward surface.

[b] Small perforations.

[c] Attacked in cleaning.

in the group III, high-phosphorus steels. Steels very low in copper corroded at high rates. The first few hundredths per cent copper had a big effect in lowering weight losses. More copper was considerably less effective. At 3.5 yr there seemed to be little merit in more than 0.1 per cent copper. At 15.5 yr it was clear that there was continued improvement with up to 1 per cent copper or more.

A similar plot of the group II, low-phosphorus steels would appear much the same as Fig. 3(*a*) except that all the curves would show somewhat higher weight losses. This shows that phosphorus is beneficial in copper steels. Similar plots of the group I and group IV steels would show comparatively high corrosion rates for the relatively pure materials of group I and comparatively low rates for the more complex steels of group IV. It is thought that this was due largely to differences in manganese content.

Figure 3(*b*) shows the effect of manganese in low phosphorus steels containing 0.3 per cent copper. Manganese additions were beneficial in these steels at the marine Kure Beach location. As described previously, manganese was also beneficial at the marine Block Island location but was not nearly as effective at the industrial Bayonne location (**4**).

Figure 3(*c*) shows the effect of nickel content in the steels of group VII. All these steels contained 0.03 or more per cent copper, so the intercepts at zero per cent nickel were taken from a plot of the group II low-phosphorus steels. A little nickel brought about a marked improvement, and there was continued improvement with larger amounts of nickel. The curve at 15.5 yr was steeper than the

TABLE IV.—AVERAGE CORROSION RATES FROM WEIGHT LOSSES, mil per year.

Steel	Kure Beach (800-ft site), N. C.			Block Island, R. I.			Bayonne, N. J.		
	0 to 7.5 yr	0 to 15.5 yr	7.5 to 15.5 yr	0 to 9.1 yr	0 to 17.1 yr	9.1 to 17.1 yr	0 to 9.1 yr	0 to 18.1 yr	9.1 to 18.1 yr
No. 45	...	...	...	...	...	...	1.71	...	...
No. 63	1.29	1.40	1.51	...	...	...	0.89	0.61	0.31
No. 54	1.11	0.97	0.85	...	...	...	0.75	...	...
No. 55	1.01	0.84	0.67	...	...	...	0.60	...	...
No. 10	1.33	...	...	...	...	...	0.98	...	...
No. 31	1.02	...	...	...	...	...	0.83	...	...
No. 5	1.11	...	...	...	...	...	0.87	...	...
No. 57	0.97	0.76	0.57	...	...	...	0.59	...	...
No. 6	0.94	0.75	0.56	...	...	...	0.57	...	...
No. 41	0.93	0.76	0.60	1.30	...	...	0.57	0.41	0.25
No. 43	0.92	0.75	0.59	1.23	...	...	0.58	0.41	0.24
No. 64	0.91	0.72	0.54	1.20	...	...	0.56	0.40	0.23
No. 40	0.93	0.77	0.62	1.43	...	...	0.56	0.40	0.24
No. 58	0.90	0.70	0.51	1.26	...	...	0.55	0.38	0.21
No. 42	0.88	0.71	0.56	1.12	...	...	0.49	0.38	0.27
No. 56	0.90	0.71	0.52	1.19	...	...	0.55	...	...
No. 7	0.89	0.67	0.46	...	...	...	0.52	...	...
No. 8	0.73	0.56	0.41	...	...	...	...	...	...
No. 26	1.19	...	...	...	...	...	...	...	...
No. 11	0.91	0.68	0.47	...	...	...	0.70	...	...
No. 2	0.73	0.58	0.45	0.88	0.83	0.77	0.48	0.33	0.19
No. 12	0.81	0.62	0.44	0.91	...	...	0.50	...	...
No. 16	0.73	0.55	0.38	0.79	...	...	0.46	...	...
No. 3	0.69	0.54	0.29	0.78	0.64	0.48	0.40	0.28	0.17
No. 4	0.61	0.44	0.28	...	...	...	...	...	...
No. 18	0.72	0.53	0.35	0.88	...	...	0.54	...	...
No. 19	0.71	0.55	0.40	0.79	...	...	0.62	...	...
No. 17	0.67	0.53	0.40	0.72	...	...	0.46	...	...
No. 66	0.71	0.54	0.38	0.77	0.72	0.66	0.56	0.35	0.14
No. 53	0.58	0.44	0.32	0.68	...	...	0.44	0.28	0.11
No. 67	...	...	...	0.42	0.35	0.28	0.44	...	...
No. 68	...	...	...	0.42	0.35	0.28	0.38	...	...
No. 154	...	...	...	0.35	0.29	0.22	0.24	...	...
No. 97	0.31	...	...	0.43	...	...	0.33	0.19	0.06
No. 13	0.31	0.21	0.11	0.36	...	...	0.19	0.11	0.03
No. 118	0.91	0.69	0.49	1.21	1.16	1.10	0.52	0.37	0.22
No. 120	0.86	0.66	0.47	1.17	1.06	0.94	0.49	0.35	0.22
No. 38	0.59	0.45	0.32	0.67	0.52	0.35	0.45	0.29	0.12
No. 121	0.67	0.48	0.30	0.77	0.64	0.48	0.39	0.27	0.14
No. 48	0.53	0.38	0.25	0.60	0.50	0.37	0.36	0.24	0.11
No. 32	0.88	0.70	0.53	...	...	...	0.67	...	...
No. 27	0.69	0.52	0.35	0.82	...	...	0.54	...	...
No. 33	0.85	0.66	0.49	...	...	...	0.60	...	...
No. 34	0.58	0.46	0.35	0.70	...	...	0.45	...	...
No. 28	0.55	0.41	0.27	0.59	...	...	0.44	...	...
No. 147	...	...	...	0.53	...	...	0.40	...	...
No. 46	0.43	0.41	0.39	0.46	...	...	0.39	...	...
No. 35	0.54	0.40	0.27	0.56	...	...	...	...	...
No. 20	0.42	0.30	0.18	0.45	...	...	0.35	...	...
No. 62	0.28	0.20	0.12	...	0.23	0.17[a]	0.27	0.16	0.04
No. 36	0.34	0.24	0.15	0.39	0.28	0.15	0.31	0.19	0.07
No. 80	0.46	0.34	0.23	...	...	...	0.35	...	...

TABLE IV.—(*Concluded*).

Steel	Kure Beach (800-ft site), N. C.			Block Island, R. I.			Bayonne, N. J.		
	0 to 7.5 yr	0 to 15.5 yr	7.5 to 15.5 yr	0 to 9.1 yr	0 to 17.1 yr	9.1 to 17.1 yr	0 to 9.1 yr	0 to 18.1 yr	9.1 to 18.1 yr
No. 29......	0.44	0.32	0.21	0.42	...	...	0.34	...	...
No. 51......	0.30	0.21	0.13	0.30	...	...	0.22	0.12	0.03
No. 52......	0.34	0.25	0.16	0.31	...	...	0.25	0.14	0.03
No. 37......	0.43	0.31	0.19	0.50	0.43	0.35	0.30	0.19	0.08
No. 60......	0.49	0.35	0.21	0.56	...	...	0.37	...	...
No. 148......	...	...	...	...	...	...	0.30	...	...
No. 50......	0.29	0.18	0.08	0.34	...	...	0.27	...	...
No. 39......	0.46	0.33	0.20	0.53	0.41	0.27	0.35	0.22	0.08
No. 149......	...	...	...	0.42	...	...	0.29	0.23	0.16
No. 150......	...	...	...	0.44	...	...	0.28	0.18	0.08
No. 153......	...	...	...	0.37	...	...	...	0.12	0.05[b]
No. 152......	...	...	...	0.35	...	...	...	0.15	0.07[b]
No. 151......	...	...	...	0.35	...	...	. .	0.13	0.06[b]
No. 49......	0.65	0.50	0.35	0.72	0.63	0.52	0.42	0.29	0.16
No. 16-1......	0.73	0.52	0.32	0.80	...	...	0.40	...	...
No. 59......	0.47	0.34	0.22	0.50	0.39	0.27	0.33	0.23	0.12
No. 47......	0.43	0.30	0.18	0.47	...	...	0.32	...	...
No. 14......	0.42	0.30	0.18	0.48	...	...	0.30	0.18	0.06
No. 119......	0.77	0.59	0.42	0.94	0.89	0.73	0.44	0.30	0.16
No. 65......	0.51	0.37	0.23	0.59	0.51	0.42	0.34	...	...

[a] Average corrosion rate 5.0 to 17.1 yr.

[b] Average corrosion rate 7.1 to 18.1 yr.

curve at 3.5 yr, showing that the beneficial effect of alloying increased with time. The high nickel steels had quite low weight losses.

Figure 3(*d*) shows the effect of chromium in high phosphorus steels containing about 0.4 per cent copper. The intercepts at zero per cent chromium were read off Fig. 3(*a*) at 0.4 per cent copper. It is clear that chromium has a beneficial effect in decreasing weight losses. The beneficial effect of chromium was particularly pronounced on long exposure.

The data do not permit a clear evaluation of the effects of other elements added individually to the steels or of the combined effect of several elements added simultaneously. As discussed previously (4) molybdenum did not seem particularly valuable in groups VI, IX, or XIII, but on the other hand the nickel-chromium-molybdenum steels of group X had quite low weight losses. Silicon generally seemed without much effect, although it probably was useful in the group V steels. The effects of composition have been discussed in considerable detail in the literature (5). It can be concluded that copper, phosphorus, nickel, and chromium alone or in appropriate combinations are the most effective elements in decreasing the atmospheric corrosion of steels.

RESULTS AT BLOCK ISLAND

Five specimens of each steel were exposed at Block Island. These were removed individually at different time periods chosen on the basis of their composition and thickness. They were then cleaned, weighed, and pit depths measured. The weight losses are given in Table II and the pit depths in Table III. The new data at 17.1 yr form logical extensions of the curves previously plotted through 9 yr (**4**). Both the rate of corrosion and the rate of pitting continued to decrease somewhat with time. The weight loss data were quite similar to those at Kure Beach except that the corrosion was somewhat more severe, particularly on the poorer steels. The effects of composition were similar (**4**).

Some of the steels again proved to have remarkable durability. Taking the 5 per cent nickel steels as an example, at 17 yr the minimum loss in thickness as measured by flat micrometers was 0.0 mil, the average penetration from each surface as calculated from weight losses was 4.4 mils, and the maximum penetration of the deepest pits as given in Table III was 12.4 mils. For all the steels, pit depths at 17 yr ran two to three times greater than the penetration calculated from weight losses. Pitting factors (ratio of pit depths to average corrosion) tended to decrease somewhat with time.

RESULTS AT BAYONNE

Five specimens of each steel were also exposed at Bayonne. They were handled in the same manner as the Block Island specimens. Weight losses are in Table II and pit depths in Table III. The new data at 18.1 yr again form logical extensions of the curves previously published through 9 yr (**4**).

As previously reported (**3 4**) the behavior of the steels in the industrial Bayonne atmosphere was somewhat different than in the marine atmospheres. The rust coatings were more protective and the rates of corrosion and the rates of pitting decreased to lower values. While the major effects of alloying were similar there were minor dfferences. Copper and chromium additions were somewhat more beneficial at Bayonne and manganese additions more helpful at the marine locations (**4**).

The better low alloy steels were even more durable at Bayonne than at the marine locations. Again taking the 5 per cent nickel steels as an example, at 18 yr the minimum loss in thickness as measured with flat micrometers was less than 1 mil, the average penetration from each surface as calculated from weight losses was 3.2 mils, and the maximum penetration of the deepest pits as given in Table III was 10.6 mils. On long exposure, pitting factors tended to run a little larger at Bayonne than at Block Island.

DURABILITY OF BETTER STEELS

In Table IV are given the average corrosion rates from weight losses, expressed as mils per year, for all steels for which data are available for exposures of 7.5 yr or longer. At Kure Beach the corrosion rates are calculated from zero to 7.5 yr, from zero to 15.5 yr, and from 7.5 to 15.5 yr. With one exception (steel No. 63), the average corrosion rate for 15.5 yr is less than for 7.5 yr, and the rate for 7.5 to 15.5 yr is smaller still. This latter rate, or something even less, is the rate which would be expected to apply to longer exposures. Similar data are in the table for the Block Island and Bayonne locations.

At Kure Beach the average corrosion rate from 7.5 to 15.5 yr of the ten best steels in the test (Nos. 13, 20, 62, 36, 51, 52, 37, 50, 47, and 14) is 0.15 mil per yr. The average corrosion rate for the copper steels of group II (Nos. 57, 6, 41, 43, 64, 40, 58, 42, 56) is 0.56 mil per yr. The corrosion rate of carbon steels would be higher than this, and of unalloyed open-hearth irons much higher, but reliable long-time figures are not available because most of these materials corroded away early in the tests.

At Bayonne the average corrosion rate from 9.1 to 18.1 yr for the low-alloy steels in the first group above for which data are available (Nos. 13, 62, 36, 51, 52, 37, and 14) is 0.05 mil per yr. For these same steels the average depth of the deepest pits was 9.8 mils at 9.1 yr, and from 9.1 to 18.1 yr the pits were deepening at an average rate of 0.17 mil per yr.

It is of interest to compare the long-time corrosion rates of good low-alloy steels with those of nonferrous metals. Indications are that in industrial atmospheres these rates are of the same order of magnitude. For example, in some 20-yr tests at New York City, which has an industrial atmosphere similar to Bayonne, the corrosion rate of copper was 0.05 mil per yr, of 70–30 brass 0.10, of Monel nickel-copper alloy 0.06, of zinc 0.22, of nickel 0.14, and of tin 0.05 mil per yr (**6**). In marine atmospheres, the comparisons are not as favorable, as many nonferrous metals corrode less there than in industrial atmospheres (**6**) while steels corrode more.

It is clear that bare, reasonably thick specimens of good low-alloy steels will have good durability boldly exposed outdoors in most types of atmospheres. It should be pointed out that shelter, crevices, undrained areas, and other factors can increase the corrosion (**3,7,8**). The use of such low-alloy steels in combination with protective coatings can be particularly effective (**9,10**).

Acknowledgment:

Numerous individuals were involved in getting these tests started, in carrying them along, and in obtaining the results. The author wishes to acknowledge the help of all these people.

REFERENCES

(1) F. L. LaQue, "Corrosion Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 51, p. 495 (1951).

(2) H. R. Copson, "A Theory of the Mechanism of Rusting of Low Alloy Steels in the Atmosphere," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 554 (1945).

(3) H. R. Copson, "Factors of Importance in the Atmospheric Corrosion Testing of Low-Alloy Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 591 (1948).

(4) H. R. Copson, "Atmospheric Corrosion of Low-Alloy Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, p. 1005 (1952).

(5) H. J. French and F. L. LaQue, "Alloy Constructional Steels," Am. Soc. for Metals (1942).

(6) Symposium on Atmospheric Corrosion of Nonferrous Metals (1955). (Issued as separate publication *ASTM STP No. 175*.)

(7) N. B. Pilling and W. A. Wesley, "Atmospheric Durability of Steels Containing Nickel and Copper—Additional Exposure Data," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 610 (1948).

(8) H. R. Copson, "Effect of Specimen Shape on Corrosion in the Atmosphere," *Corrosion*, Vol. 7, p. 335 (1951).

(9) H. R. Copson and C. P. Larrabee, "Extra Durability of Paint on Low-Alloy Steels," ASTM BULLETIN, No. 242, Dec. 1959, p. 68 (TP 228).

(10) F. L. LaQue and J. A. Boylan, "Effect of Composition of Steel on the Performance of Organic Coatings in Atmospheric Corrosion," *Corrosion*, Vol. 9, p. 237 (1953).

DISCUSSION

Mr. F. J. Radd.[1]—I would like to ask the author as to his interpretation of the mechanism of the copper protectiveness in the atmospheric tests.

Mr. H. R. Copson (*author*).—It is probable that copper in the steel has more than one effect in reducing atmospheric corrosion. Steels contain some sulfur, and this sulfur content has the effect of increasing the corrosion rate in the atmosphere. One function of copper is to tie up the sulfur in the steel as a copper sulfide. It does not take much copper to do this, and this may account for the major initial effect of a little bit of copper. When you get to higher copper contents, and to higher nickel contents, then the subject gets more complicated. The steels build up protective rust coatings, which slow down the corrosion. We made some analyses years ago of the rust coating on steels and found that the amount of sulfate which is tied up in the rust increases with the alloy content (reference (2) of the paper). If you increase the nickel content, you get more sulfate in the rust. We interpret this as meaning that one function of some alloying elements is to render sulfate corrosion products insoluble. In an industrial atmosphere there is a lot of SO_2 around to form iron sulfates along with iron hydroxide. The iron sulfates hydrolyze, they are somewhat soluble, they may be hygroscopic, and they may wash away leaving the rust porous. All this increases corrosion. An addition to the steel which makes the corrosion products more insoluble helps form a more protective and less porous rust coating. Additions of copper, nickel, and some other elements make the rust coatings more insoluble, more adherent, thinner, and definitely more protective.

Mr. G. M. Batchelder.[2]—I would like to ask the author if Kure Beach and Block Island are considered severe marine corrosion atmospheres. If not, has any work been done on what would be considered a severe atmosphere?

Mr. Copson.—The severity of a marine location varies directly with nearness to the ocean. If the location is right on a beach where there is a lot of surf and spray and the wind direction is right, then a lot of salt is carried into the air and corrosion can be very severe. As the location is moved away from the beach the corrosion rate decreases quite rapidly. These particular tests at Kure Beach were carried out at a site which is about 800 ft from the ocean. Other tests at Kure Beach have been conducted on the beach roughly 80 ft from the ocean. Here the corrosion rate of steel is much higher. The effect is bigger on the purer materials. The low-alloy steels show a greater comparative advantage at the 80-ft site than at the 800-ft site. A comparison of the relative corrosivity of different atmospheres has shown that Kure Beach can be considered a severe marine atmosphere (reference (1) of the paper). The Block Island location was a little more corrosive than the 800-ft lot at Kure Beach. It is very hard to define a new unknown atmosphere. The best way to compare this with another location is to run tests on control specimens.

---

[1] Research Group Leader, Continental Oil Co., Ponca City, Okla.

[2] Research Assistant Professor, Engineering Experimental Station, University of New Hampshire, Durham, N. H.

EFFECTS OF ALLOYING ADDITIONS ON HOT CRACKING OF AUSTENITIC CHROMIUM-NICKEL STAINLESS STEELS

By Frederick C. Hull[1]

Synposis

The quantitative effects of various elements on hot cracking of cast austenitic chromium-nickel stainless steels have been determined by means of the Cast Pin Tear test. Nitrogen, manganese, molybdenum, tungsten, and chromium decrease the susceptibility to hot cracking. On the basis of equal per cent by weight, cobalt, vanadium, aluminum, nickel, tantalum, copper, silicon, columbium, titanium, carbon, hafnium, zirconium, and boron cause increasing amounts of cracking, in the order listed. These data may be used to design alloys with improved resistance to hot cracking.

Many types of cracking problems are encountered in both the fabrication and application of welded stainless steels at elevated temperatures. Cracks may occur in three locations (in the weld deposit, in the heat-affected zone of the base metal, and in the base metal) and at three different times (during welding or the initial cooling, during subsequent solution annealing or stress-relieving treatment, and during long-time high-temperature service). The present paper is concerned exclusively with the effects of alloying elements on cracking during solidification, such as the hot cracking of as-deposited weld metal and the hot tearing of castings in the foundry. It is hoped that the optimum compositions for resisting cracking in the as-cast condition may also prove to be less susceptible to the other types of cracking encountered.

Numerous qualitative statements appear in the literature on the beneficial or detrimental effects of various alloying additions and of delta ferrite on weld-bead cracking. There are a few papers in which cracking is expressed quantitatively, but even in these cases the results from one method of testing to another are difficult to correlate. Moreover, comparisons for the same method cannot always be made safely because of variations at different laboratories in preparation of electrodes or the base metal or in techniques of welding. In order to establish the quantitative effects of many different alloying elements, a large number of steels must be tested under comparable conditions.

The Cast Pin Tear (CPT) test (**1**)[2] is a rapid and inexpensive test that produces cracks in chill-cast pins as they freeze and cool under conditions intended to simulate the restraint imposed during welding of heavy sections. In iron-chromium-nickel alloys, compositions with the least susceptibility to hot cracking as determined by the CPT test

---

* Presented at the Sixty-third Annual Meeting of The Society, June 27–July 1, 1960.

[1] Advisory Metallurgist, Westinghouse Research Laboratories, Pittsburgh, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

coincide with a region in which 7 to 10 per cent delta ferrite occurs in the as-cast structure. Since this finding agrees with general welding experience, predictions based on the CPT test should be valid for weldability studies. By using this test, it was possible in a period of one year to evaluate quantitatively the effects of 18 alloying elements on the hot-cracking tendencies of chromium-nickel stainless steels. These data should be useful in the development of stainless steel electrodes having improved weldability and of casting compositions having greater resistance to hot tearing.

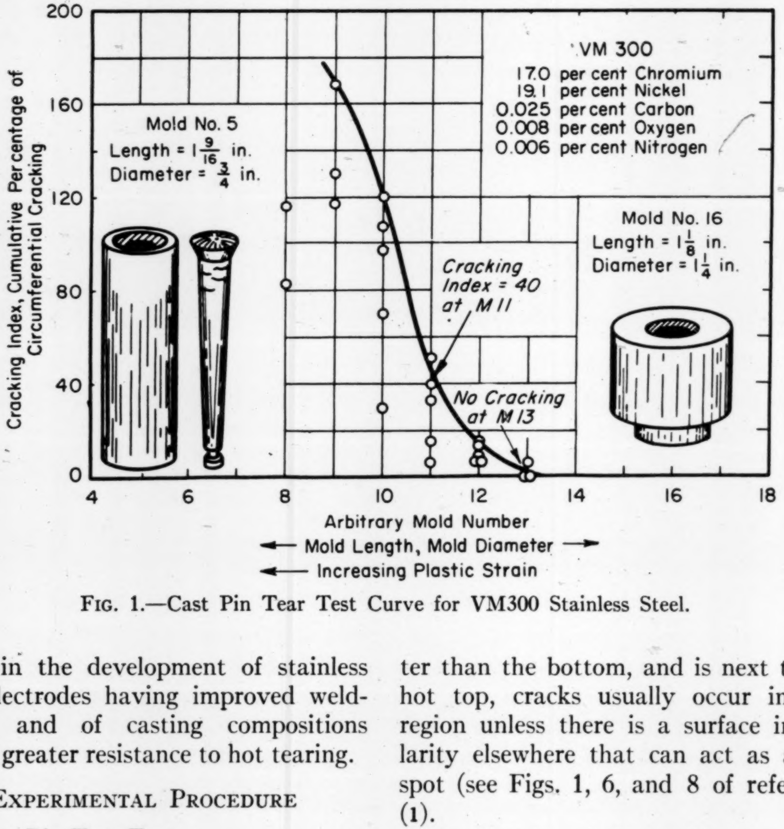


FIG. 1.—Cast Pin Tear Test Curve for VM300 Stainless Steel.

EXPERIMENTAL PROCEDURE

The Cast Pin Tear Test:

The CPT test for susceptibility to hot cracking is carried out by levitation melting 19-g samples of an alloy in an argon atmosphere and casting the melts as about $\frac{1}{4}$-in. diameter tapered pins in a series of copper molds. Restraints at the ends of the pin impose a tensile strain on the sample as a result of the thermal contraction of the casting and the thermal expansion of the mold. Mold length would not be an important variable if the temperature of the pin were uniform, but there is usually a hot spot at which the strain can be localized, causing the severity of the strain to increase with the length of the mold. Since the top of the pin is poured last, is larger in diameter than the bottom, and is next to the hot top, cracks usually occur in this region unless there is a surface irregularity elsewhere that can act as a hot spot (see Figs. 1, 6, and 8 of reference (**1**).

The lengths of the molds vary from $1\frac{1}{8}$ to $2\frac{3}{4}$ in. and their outside diameters from $\frac{3}{4}$ to $1\frac{1}{4}$ in. From an examination of the surface cracking of the pin at a magnification of × 30, the cumulative percentage of circumferential cracking (cracking index) is determined and is then plotted against arbitrary mold numbers to provide a measure of the susceptibility of the alloy to hot cracking. In the typical CPT test curve illustrated in Fig. 1, the long, slender molds (which have the smaller assigned numbers) produce severe cracking, whereas the short, heavy-walled molds (which have the larger numbers) crack the pin less severely or not at all. The chance variations in hot-spot conditions from one pin to another are probably responsible for the considerable differences in the amounts of cracking observed. The cracking curve was drawn through the points of the greatest amount of cracking for each mold, except for occasional high values that could not be reproduced.

Two arbitrary points are taken from the CPT test curve as representing the susceptibility of the alloy to hot cracking—the mold numbers for cracking indices of 0 and 40. Throughout the figures of this paper, these mold numbers are plotted against composition to illustrate the effects of alloying additions on cracking. The "no cracking" curve is usually parallel to and two or three numbers greater than the curve for "cracking index = 40;" but since it frequently exhibits more scatter, it is preferred to make comparisons between alloys on the latter basis, as is done in the case of the two summary curves.

The susceptibility to hot cracking for each composition level of each alloying element studied was determined by CPT test curves of the type shown in Fig. 1. Since space does not permit the reproduction of over 100 such curves, it should be understood that each point in Fig. 5 through Fig. 15 represents the cracking results from 5 to 23 individual castings with an average of eleven tests for each composition.

Preparation of Alloys:

The composition ranges of the commercial steels of the AISI 300 series are such that many of the as-cast alloys contain delta ferrite. The effects of alloying additions on hot cracking in such a base would be primarily a measure of the effects of the elements on the structure (that is, per cent of delta ferrite) rather than an evaluation of the resistance to separation of the austenite grain boundaries, because the effect of ferrite is so pronounced. Other complications which could obscure the inherent effect of the alloying element can result from the formation of a second phase or the interaction of the addition with carbon and nitrogen. We therefore decided to use as the principal base composition a low-carbon, low-nitrogen alloy containing 16 per cent chromium and 20 per cent nickel that would be fully austenitic as-cast or after either hot or cold work, even with substantial additions of other elements. The investigation includes a study of the effects of various types of alloying elements: the base elements (chromium, nickel and also cobalt), interstitial elements (carbon and nitrogen), deoxidizers (manganese, silicon and aluminum), solid-solution hardeners (copper, vanadium, molybdenum and tungsten), carbide stabilizers (titanium, columbium and tantalum) and three other strong carbide- and nitride-forming elements (boron, zirconium and hafnium). The intended or analyzed compositions of the 25 and 50-lb heats are given in Table I. For some of the alloys analyses were made for impurities also, in order to establish their levels and possible significance; these results are also listed in Table I. For experimental convenience, and in order to be able to compare the effects of air and vacuum melting, four different techniques were employed in the preparation of alloys; the raw materials used in the melting step are tabulated in Table II.

TABLE I.—COMPOSITIONS OF EXPERIMENTAL STAINLESS STEELS IN PER CENT BY WEIGHT.[a]

Heat	Chromium	Nickel	Manganese	Silicon	Carbon	Nitrogen	Oxygen	Phosphorus	Sulfur	Other
No. 7136	(16)[b]	(20)	(1.2)	(0.4)	0.014	0.025	...	...	...	...
No. 7292	16.2	19.9	1.44	0.48	0.024	0.020	0.023	0.002	0.008	...
No. 7302	16.6	19.8	1.69	0.51	(0.02)	(0.02)	...	...	...	...
No. 7329	16.7	19.6	1.56	0.47	(0.02)	(0.02)	...	...	...	...
No. 7137	(16)	(20)	(0.4)	(1.25)	(0.02)	(0.02)	...	...	...	...
No. VM430	15.8	20.0	...	...	0.001	0.002	0.0045	...	...	...
No. VM200	16.1	20.0	...	...	0.0048	0.013	0.017	0.003	0.008	...
No. VM201	16.2	19.9	...	...	0.0125	0.010	0.0015	0.003	0.011	...
No. VM300	17.0	19.1	0.003	<0.03	0.025	0.0058	0.008	0.0010	0.004	...
No. VM262	16.2	20.1	...	...	0.101	0.0033	0.001	0.0005	0.0124	...
No. VM266	16.2	20.0	...	...	0.104	0.0025	0.002	0.0005	0.0105	...
No. VM253	16.3	20.1	...	...	0.124	0.0027	0.003	0.0006	0.0078	...
No. 6872	(16)	(20)	(1.2)	(0.4)	0.002	(0.03)	...	...	...	...
No. 6874	(16)	(20)	(1.2)	(0.4)	0.002	(0.03)	...	...	...	...
No. 6875	(16)	(20)	(1.2)	(0.4)	0.048	(0.03)	...	...	...	...
No. 6876	(16)	(20)	(1.2)	(0.4)	0.13	(0.03)	...	...	...	...
No. 6877	(16)	(20)	(1.2)	(0.4)	0.16	(0.03)	...	...	...	...
No. 6903	12.2	12.0	1.36	0.47	0.031	0.050	...	...	...	...
No. 6904	12.3	10.1	3.20	0.44	0.026	0.033	...	...	...	...
No. 6905	12.2	10.1	5.03	0.50	0.031	0.035	...	...	...	...
No. 6906	10.5	12.0	1.42	1.42	0.036	0.027	...	...	...	...
No. 6907	10.5	12.1	1.37	2.58	0.028	0.020	...	...	...	...
No. 6908	12.2	10.2	1.33	0.42	0.030	0.039	...	...	...	...
No. 6909	12.3	10.3	1.46	0.40	0.032	0.045	...	...	...	...
No. 6939	12.1	10.1	1.32	0.43	0.030	0.059	...	...	...	...
No. 6940	12.3	10.0	1.28	0.45	0.034	0.062	...	...	...	...
No. 6941	10.3	12.1	1.36	1.01	0.048	0.027	...	0.010	0.015	...
No. 6942	10.2	12.0	1.39	1.96	0.038	0.028	...	...	...	...
No. 7375	16.1	19.7	3.10	0.15	0.012	0.010	...	...	...	...
No. 7055	16.5	17.1	6.47	0.39	0.025	0.029	...	...	...	...
No. VM205	16.0	20.0	1.45	...	0.012	0.016	0.0037	0.003	0.011	...
No. VM202	16.1	20.0	...	0.53	0.0053	0.010	0.014	0.003	0.007	...
No. VM208	15.0	20.3	...	...	0.0049	0.015	0.0054	0.003	0.008	0.32 aluminum
No. 7303	16.8	19.7	1.54	0.49	(0.015)	(0.02)	...	...	...	1.00 molybdenum
No. VM203	16.1	19.9	...	...	0.0013	0.011	0.027	0.003	0.007	1.51 molybdenum
No. 7049	16.4	19.2	1.29	0.41	0.025	(0.02)	...	...	...	1.95 molybdenum

No. 7304	16.5	19.3	1.53	0.44	(0.015)	(0.02)	...	...	...	1.95 molybdenum
No. 7050	16.3	19.9	1.37	0.43	0.027	(0.02)	...	...	...	2.94 molybdenum
No. 7305	16.7	19.7	1.55	0.50	(0.015)	(0.02)	...	...	...	2.93 molybdenum
No. 7306	16.8	19.8	1.56	0.51	(0.015)	(0.02)	...	...	...	4.17 molybdenum
No. 7307	17.2	18.5	1.61	0.52	(0.015)	(0.02)	...	...	...	5.38 molybdenum
No. 7060	16.3	20.0	1.29	0.45	0.023	(0.02)	...	...	...	0.17 vanadium
No. VM414	16.4	19.9	...	...	0.0013	0.0042	0.0016	...	...	1.01 vanadium
No. VM416	16.8	20.1	...	...	0.0035	0.0031	0.0031	...	...	2.09 vanadium
No. VM419[c]	14.8	19.9	...	...	0.0014	0.0030	0.0033	...	...	4.65 vanadium
No. 7051	16.2	20.1	1.26	0.43	0.030	(0.02)	...	...	...	3.02 molybdenum, 2.05 copper
No. VM420	16.0	20.0	...	...	0.0028	0.0018	0.0034	...	...	4.69 tungsten
No. 7053	19.6	23.7	1.61	0.59	0.033	0.014	...	...	...	0.42 titanium
No. 7054	16.5	19.9	1.26	0.49	0.028	(0.02)	0.010	...	...	0.54 titanium
No. VM204	16.3	20.0			0.012	0.015	0.0032	0.003	0.011	0.54 titanium
No. 7177	(17)	(20)	(1.3)	(0.4)	(0.02)	(0.02)	0.004	...	...	0.75 titanium
No. BT69	(16)	(20)	(1.3)	(0.4)	(0.05)	(0.02)	...	...	...	(0.9) titanium
No. M3533	16.5	20.3	1.36	0.50	0.050	(0.02)	0.003	0.005	0.004	0.93 titanium
No. 7102	(16)	(20)	(1.2)	(0.4)	(0.1)	(0.02)	...	...	...	(0.5) columbium
No. 7101	(16)	(20)	(1.2)	(0.4)	0.022	0.016	...	...	...	0.57 columbium
No. 7104	(16)	(20)	(1.2)	(0.4)	(0.1)	(0.02)	...	...	...	(1) columbium
No. 7103	(16)	(20)	(1.2)	(0.4)	0.031	0.020	...	...	...	1.24 columbium
No. VM265	16.3	20.0	...	...	0.004	0.0037	0.013	0.0005	0.004	1.28 columbium
No. 7052	17.2	17.2	1.39	0.72	0.039	(0.02)	...	...	...	1.72 columbium
No. 7381	16.4	20.1	1.59	0.17	0.016	0.055	...	...	...	0.16 tantalum
No. 7106	(16)	(20)	(1.2)	(0.4)	0.024	0.025	...	...	...	0.11 boron
No. 7105	(16)	(20)	(1.2)	(0.4)	0.035	0.017	...	...	...	0.20 boron
No. VM267	16.2	20.0	...	...	0.0068	0.0019	0.006	0.0005	0.0037	0.005 boron, 0.028 zirconium
No. VM264	16.5	19.5	...	...	0.0044	0.0038	0.041	0.0005	0.0071	0.08 zirconium
No. 7107	(16)	(20)	(1.2)	(0.4)	0.019	0.008	...	...	...	0.74 zirconium
No. VM206	15.8	20.2	...	...	0.0020	0.013	0.015	0.003	0.009	0.97 zirconium
No. 7058	16.3	19.8	1.33	0.49	0.032	(0.02)	...	...	...	1.57 zirconium
No. VM210	16.1	20.0	...	...	0.0019	0.019	0.0059	0.003	0.009	0.44 hafnium
No. 7059	16.0	19.5	1.37	0.43	0.036	0.17	...	...	...	...

[a] Balance iron.

[b] Values in parentheses are intended compositions.

[c] 1.8 per cent delta ferrite.

Air Melts:

Air melts were made by high-frequency induction melting in magnesia crucibles. The melting sequence was first the iron, then molybdenum (if desired), one half the nickel, chromium, and then the remaining nickel. Deoxidation was effected by adding 0.3 per cent CaSi and then additional silicon and manganese to a specified level. Reactive metals like vanadium, titanium, columbium, zirconium, and boron were stirred into the melt just before pouring to minimize losses by reaction with the crucible, the slag, or the atmosphere. An ingot weighing 25 lb was cast in a sheet-iron mold that was surrounded by sand and that had a massive copper stool and a gas-heated ceramic hot-top.

TABLE II.—RAW MATERIALS USED IN MELTING.
(Analyses in per cent by weight

Al—99 + Aluminum
B—15 to 18 boron, balance nickel
C—Graphite
Cb—Ferrocolumbium: 0.33 carbon, 6.3 silicon, 58 columbium
    Pure columbium (vacuum melts)
Cr—Electrolytic chromium: 0.01 carbon, 0.036 sulfur, 0.36 oxygen
    High-purity chromium grade A: 0.01 carbon, 0.01 oxygen (compacts)
    Ferrochromium (heats 6902-6942)
Cu—OFHC copper
Fe—Armco iron (heats 6902–6909, 6941, 6942)
    Electrolytic A101: 0.018 carbon, 0.005 phosphorus, 0.004 sulfur, 0.07 oxygen (other air melts)
    Electrolytic A104: 0.004 carbon, 0.003 phosphorus, 0.004 sulfur, 0.05 oxygen (vacuum melts, compacts)
Hf—Iodide crystal bar
Mn—99.95 electrolytic manganese
Mo—99.9 sintered bar
N—Nitrided ferrochromium
Ni—Electrolytic nickel: 99.95 nickel + cobalt (0.13 cobalt)
Si—97 + silicon
Ti—Nickel-titanium eutectic: 69 titanium
Ta—Pressed pure powder pellets
V—99.5
W—Sintered bar
Zr—Zirconium sponge (2.25 hafnium)

Vacuum Melts:

Another series of alloys weighing either 25 or 50 lb was made in a Geraetebau-Anstalt Balzers VSG25 vacuum induction-melting furnace using charge materials of higher quality and a purer magnesia crucible. Initial melting was done with only the mechanical pump running. Then hydrogen was impinged on the surface of the molten bath for 30 min. After this deoxidation treatment the diffusion pump was turned on and the pressure reduced to about 1×10^{-4} mm Hg. The heat was cycled about three times from molten to solid to accelerate the removal of gases. Melts were poured into a cast-iron mold after reactive metals were added and as soon as adequate mixing occurred.

Compacts:

Some of the investigation on the alloying effects of Cr, Ni, Mn, and Si was conducted on samples prepared by pressing 19-g cylinders of mixed high-purity raw materials and levitation melting these directly in the argon atmosphere of the CPT test chamber. This technique had the dual advantages of never exposing the melt to the possibility of contamination by refractories and minimizing the quantity of materials needed for the tests. Chemical analyses were not made of these small heats nor are they listed in Table I. The intended analyses may be judged from the appropriate figures.

Doping:

The most convenient and reliable method for determining the effects on hot cracking of small additions of an element is by doping of a master alloy. Two of the vacuum melts (VM 300 and VM 430 steels) were rolled to $\frac{5}{8}$-in. diameter bar stock for this purpose. The bars were machined to remove the surface oxide and cut to short lengths; then a small

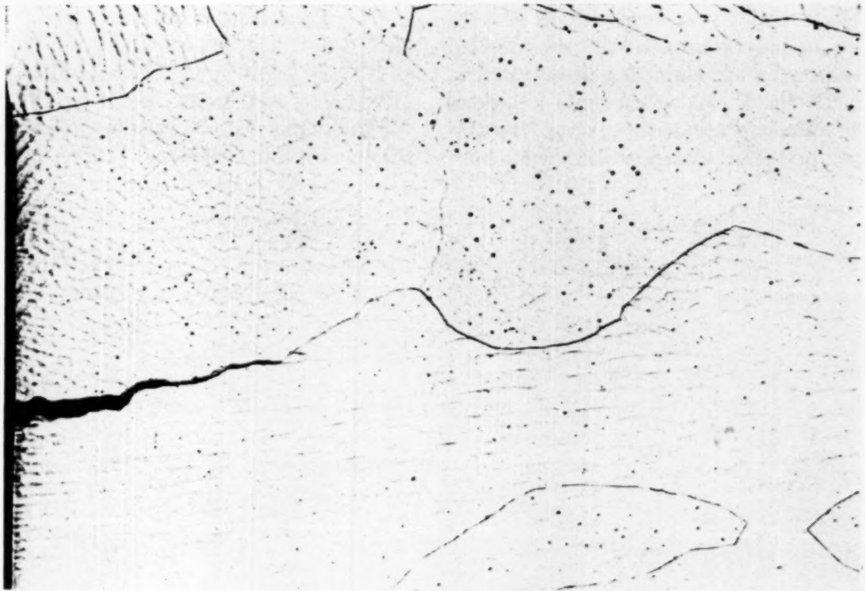


FIG. 2.—Example of Surface Hot Cracking in Heat VM420 Steel (16 per cent Chromium, 20 per cent Nickel, 4.7 per cent Tungsten) (×200).

Note smooth mobile grain boundaries.

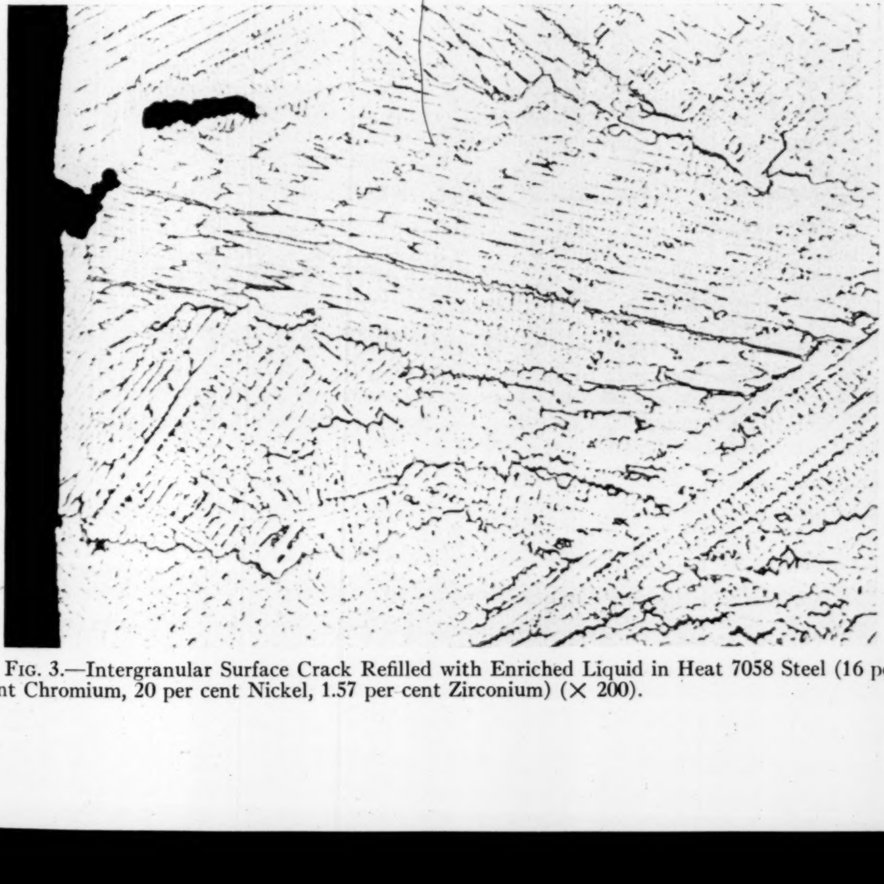


FIG. 3.—Intergranular Surface Crack Refilled with Enriched Liquid in Heat 7058 Steel (16 per cent Chromium, 20 per cent Nickel, 1.57 per cent Zirconium) (× 200).

hole was drilled in one end of each of these cylinders into which the desired amount of such elements as Al, B, Co, Cu, Hf, Ta, V, W, or Zr could be added and held in place with a plug. By this procedure the composition was kept constant, except for the intentional addition, and the amount of materials needed was small. These alloys also were not analysed and are not listed in Table I. As in the case of the compacts, the points in the figures represent aim analyses.

EXPERIMENTAL RESULTS

Nature of Cracks Produced by the CPT Test:

Much has been written about the effects of low-melting eutectics on cracking, and many hypotheses of weld-metal cracking are based on the presence of a liquid film at the grain boundaries. In the paper (1) previously referred to, it was stated that ruptures of the surface produced by the CPT test were the result of hot tearing, that is, cracking in

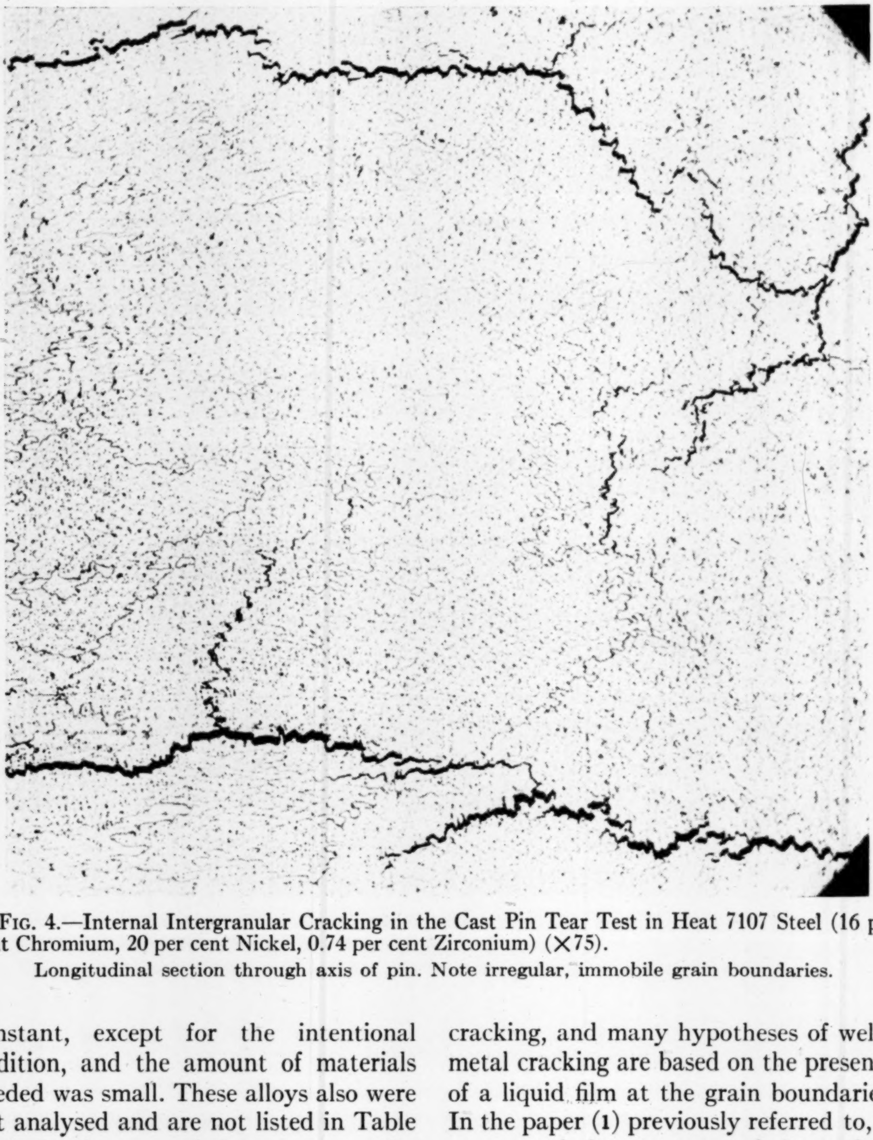


FIG. 4.—Internal Intergranular Cracking in the Cast Pin Tear Test in Heat 7107 Steel (16 per cent Chromium, 20 per cent Nickel, 0.74 per cent Zirconium) (×75).

Longitudinal section through axis of pin. Note irregular, immobile grain boundaries.

the presence of a liquid film; but a critical examination of the results accumulated since then does not fully support this conclusion. For a majority of alloying elements, and even for the exceptions at low concentrations of the addition, the interdendritic hot cracking at the surface of the pin (illustrated in Fig. 2) appears to initiate after solidification, for there are no cracks at the center of the pin where the last traces of liquid would be found.

A different situation is found in alloys containing relatively high amounts of B, Zr, Hf, and Cb. For a typical example of these cases, Fig. 3 presents evidence that surface cracks have formed and subsequently have been partially or completely filled with alloy-enriched liquid. The centers of these same specimens contain a profusion of both longitudinal and transverse cracks as depicted in Fig. 4. There is still doubt as to whether or not these interior ruptures are hot cracks or hot tears; however, the appearance of their fractured surfaces is identical to that of specimens in which only hot cracks occur, so that if a liquid is present at the time of cracking it must exist as an extremely thin film.

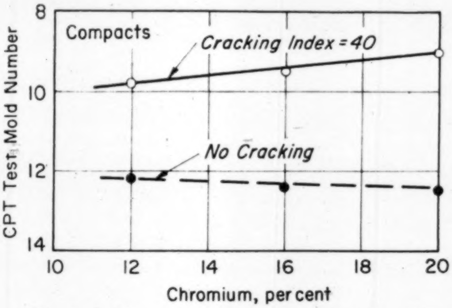


FIG. 5.—Effect of Chromium on Hot Cracking of Stainless Steels Containing 20 per cent Nickel.

The technique of reducing the apparent amount of hot tearing by providing an excess of eutectic to fill the cracks has been employed frequently in casting alloys. However, the level of the addition required to achieve this effect in stainless steels is much higher than is desirable from consideration of the mechanical properties of the material.

Effects of Alloying Additions on Hot Cracking:

As chromium is raised from 12 to 20 per cent at a 20 per cent nickel level, or as nickel is lowered from 25 to 15 per cent at a 16 per cent chromium level, the amount of hot cracking, as indicated by the line for a cracking index of 40, decreases by about one CPT test mold number as shown in Figs. 5 and 6. All of these alloys are fully austenitic as cast. When cobalt is added to the base composition, there is no appreciable change in hot cracking. For 0, 1, and 4 per cent cobalt levels the mold numbers for a cracking index of 40 are 11.0, 11.0, and 10.8, respectively.

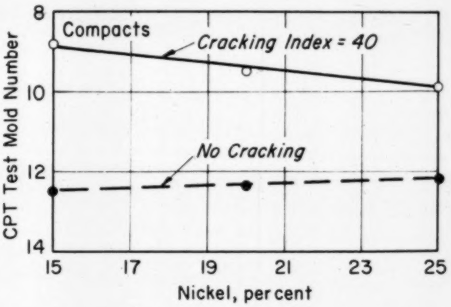
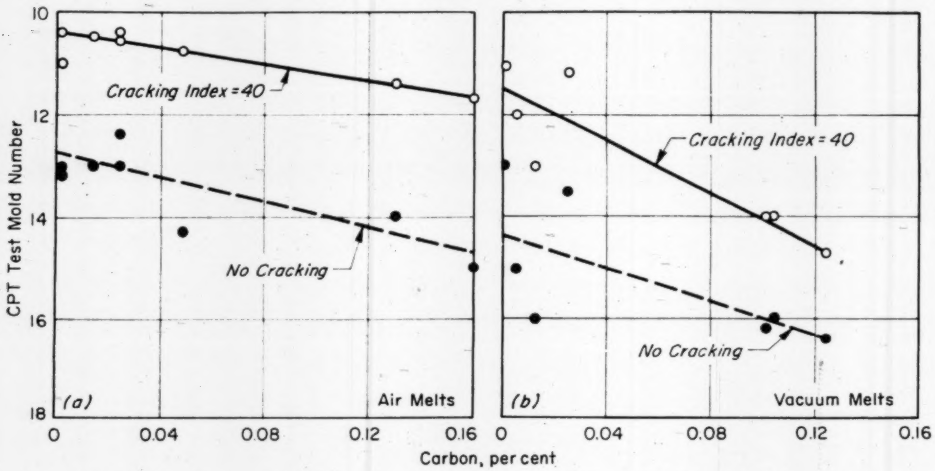


FIG. 6.—Effect of Nickel on Hot Cracking of Stainless Steels Containing 16 per cent Chromium.

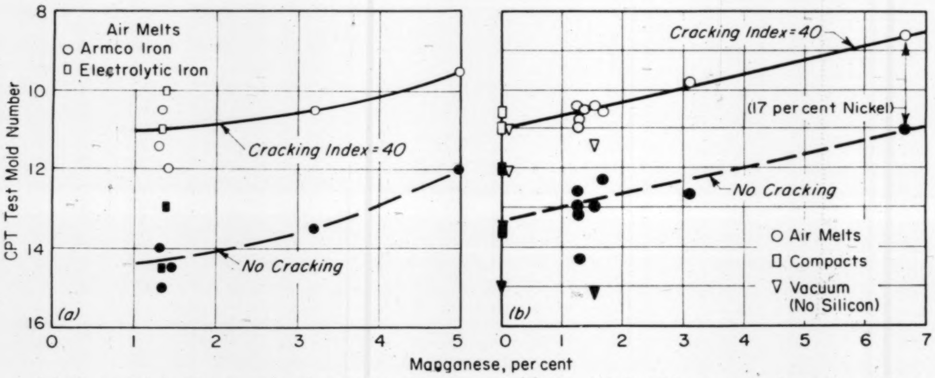
It will be noted by comparing Figs. 7(*a*) and 7(*b*) that there is a considerable difference between the cracking of air melts and vacuum melts containing carbon, although the effect of carbon *per se* on hot cracking is probably the same in either case. It is believed that the higher susceptibility of the vacuum melts is caused by reduction of oxide impurities in the crucible which then dissolve in the melt; this occurs to a greater extent at reduced pressure than at atmospheric pressure. In contrast to the increase in cracking caused by carbon, 0.17 per cent nitrogen is beneficial, on the basis of 13 tests which gave a mold number of 10.2 for a cracking index of 40 for this composition.



(*a*) Containing 16 per cent chromium, 20 per cent nickel, 1.2 per cent manganese, and 0.4 per cent silicon.

(*b*) Vacuum melted containing 16 per cent chromium and 20 per cent nickel.

FIG. 7.—Effect of Carbon on Hot Cracking of Stainless Steels.

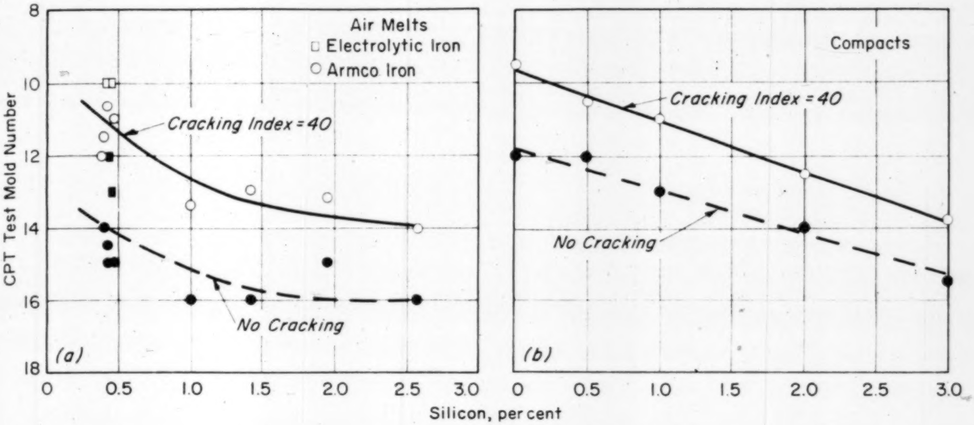


(*a*) Containing 12 per cent chromium, 10 to 12 per cent nickel, and 0.5 per cent silicon.

(*b*) Containing 16 per cent chromium, 20 per cent nickel, and 0.5 per cent silicon.

FIG. 8.—Effect of Manganese on Hot Cracking of Stainless Steels.

Substantial amounts of manganese (the data extend only up to 5 and 7 per cent manganese) decrease susceptibility to hot cracking and up to 3 per cent silicon increases it. These relationships apply at both the levels of 10 to 12 per cent chromium: 10 to 12 per cent nickel and 16 per cent chromium: 20 per cent nickel (Figs. 8 and 9). Aluminum increases cracking only very slightly up to 0.2 per cent but more rapidly at higher levels (Fig. 10(*a*)).



(a) Containing 10 to 12 per cent chromium, 12 per cent nickel, and 1.3 per cent manganese.

(b) Containing 16 per cent chromium and 20 per cent nickel.

FIG. 9.—Effect of Silicon on Hot Cracking of Stainless Steels.

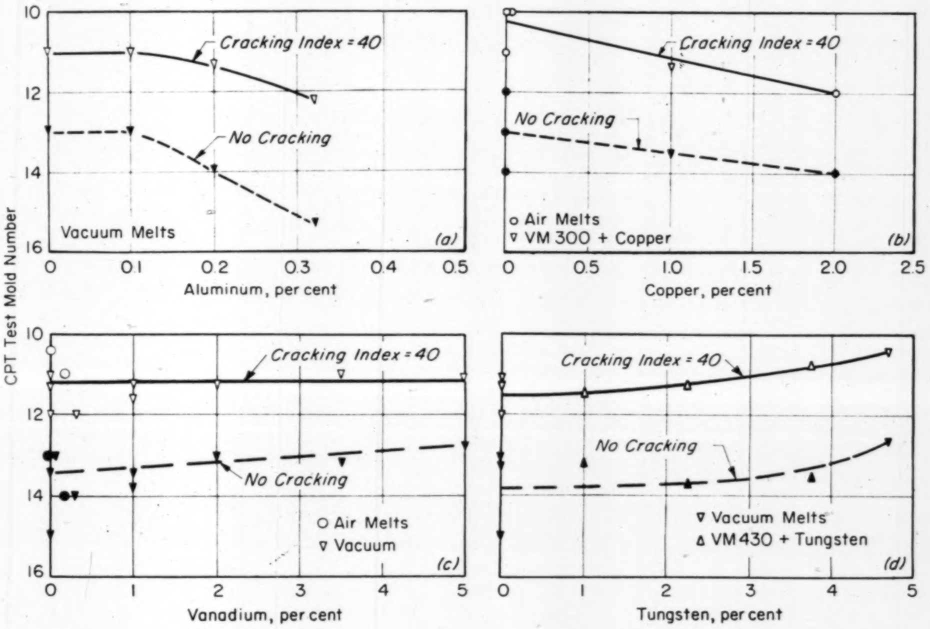


FIG. 10.—Hot Cracking of Stainless Steels Containing 16 per cent Chromium and 20 per cent Nickel.

Copper up to 2 per cent increases the amount of hot cracking (Fig. 10(*b*)). Since it is not a potent solid-solution hardener, there is no justification for copper additions in stainless steel except to obtain age hardening. In Fig. 10(*c*) it

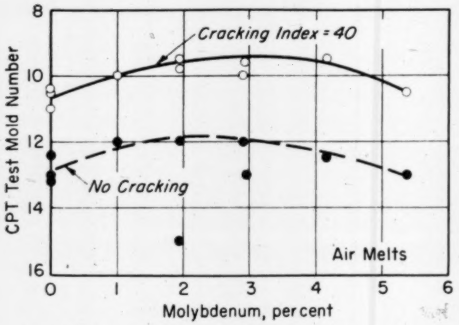
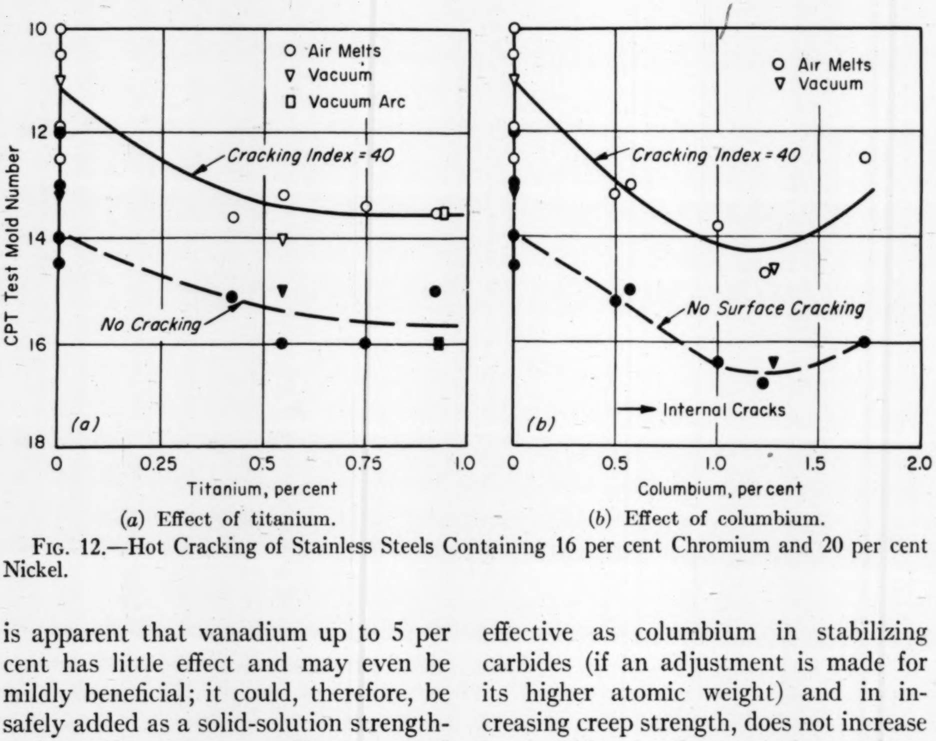


FIG. 11.—Effect of Molybdenum on Hot Cracking of Stainless Steels Containing 16 per cent Chromium, 20 per cent Nickel, 1.4 per cent Manganese, and 0.4 per cent Silicon.



(a) Effect of titanium.

(b) Effect of columbium.

FIG. 12.—Hot Cracking of Stainless Steels Containing 16 per cent Chromium and 20 per cent Nickel.

is apparent that vanadium up to 5 per cent has little effect and may even be mildly beneficial; it could, therefore, be safely added as a solid-solution strengthener or as a scavenger. Tungsten and molybdenum, which are among the most effective elements for increasing creep strength, actually decrease susceptibility to hot cracking appreciably when added in the amounts of about 5 per cent and 3 per cent, respectively (Figs. 10(*d*) and 11).

In the AISI 321 and 347 steels, titanium and columbium were added initially as carbide stabilizers to prevent intergranular corrosion. When the same alloys were found to have much higher strength at elevated temperatures than type 304 stainless steel, they were also widely used for steam power-plant and nuclear-power applications, especially the steel containing columbium. Figures 12(*a*) and 12(*b*) show that these two elements are particularly active in increasing hot-cracking susceptibility. On the other hand, tantalum, which is as effective as columbium in stabilizing carbides (if an adjustment is made for its higher atomic weight) and in increasing creep strength, does not increase hot cracking nearly as much as columbium (Fig. 13). Therefore, a tantalum-stabilized steel should have most of the advantages of a columbium-stabilized steel plus greatly improved resistance to cracking. On the negative side are (1) the greater cost of tantalum, (2) its higher cross-section for capture of neu-

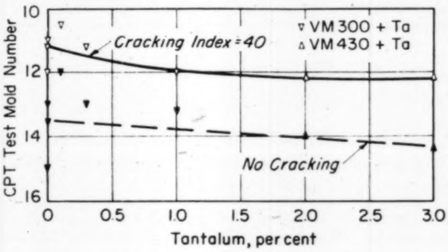


FIG. 13.—Effect of Tantalum on the Hot Cracking of Stainless Steel Containing 16 per cent Chromium and 20 per cent Nickel.

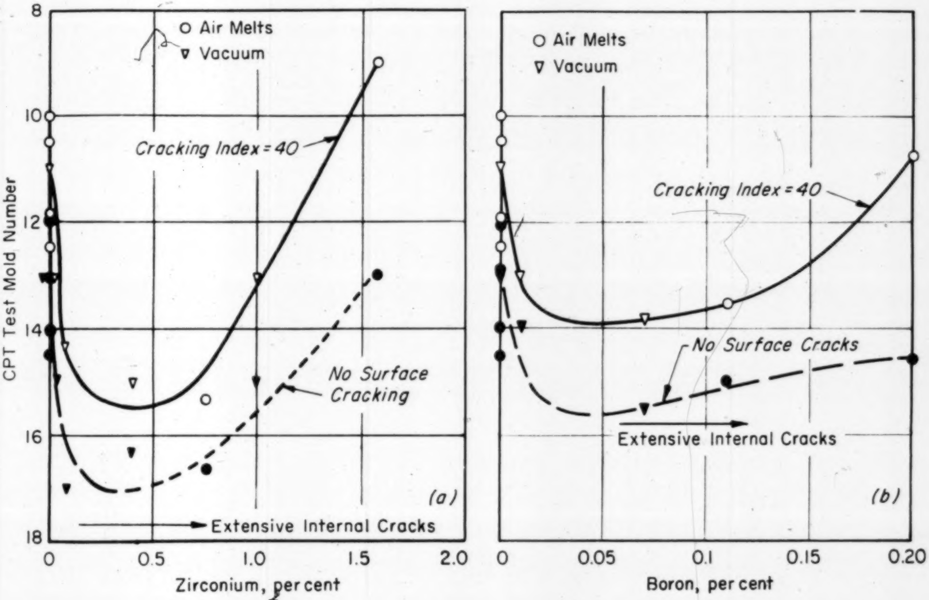


FIG. 14.—Hot Cracking of Stainless Steels Containing 16 per cent Chromium and 20 per cent Nickel.

trons which would prevent the use of tantalum in steels for nuclear-power applications, and (3) the relatively high loss of tantalum that may occur during melting or welding.

Many stainless steels and high-temperature alloys are currently being produced with additions of boron and zirconium to increase strength, creep ductility, and hot workability. In order to determine their probable effects on weldability, CPT tests were run on steels containing these elements as well as hafnium, because of its similarity to zirconium. In the ranges investigated, these three elements produce the most extensive cracking of all the additions on which quantitative data have been obtained (Figs. 14 and 15), although the minimum additions examined are considerably greater than the amounts encountered in most commercial steels.

The amounts of surface cracking in columbium-, boron- and zirconium-bearing alloys pass through maxima at 1.2 per cent columbium, 0.04 per cent boron, and 0.4 per cent zirconium, beyond which the cracking diminishes. This is only an apparent improvement, however, for when the pins of the higher alloy content are sectioned and examined metallographically, they are found to contain an abundance of internal cracks

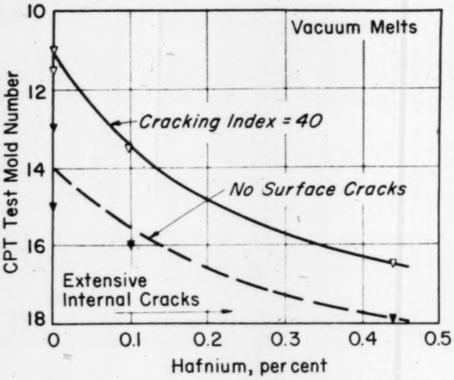


FIG. 15.—Effect of Hafnium on Hot Cracking of Stainless Steels Containing 16 per cent Chromium and 20 per cent Nickel.

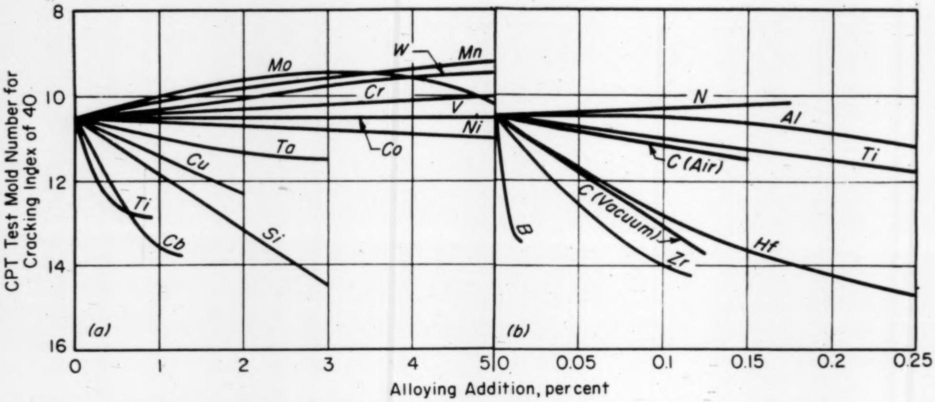


FIG. 16.—Effects of Various Elements on Hot Cracking of 16 per cent Chromium - 20 per cent Nickel Stainless Steels.

(Fig. 4). High contents of hafnium and, to a lesser extent, silicon also promote internal grain-boundary cracking.

The results of all these tests have been summarized in two curves to facilitate quantitative comparisons between the effects of different alloying elements. The beneficial additions are easily distinguished from the detrimental ones; moreover, it is fairly clear what composition limits should be specified for each element to achieve maximum resistance to hot cracking. Figure 16(*a*) includes those elements added in amounts ranging from about 0.5 to 5 per cent, whereas Fig. 16(*b*) covers the range from about 0.01 to 0.25 per cent. (The data for air melts, vacuum melts, and compacts were normalized by raising or lowering them, as necessary, to pass through a mold number of 10.5 at zero content of the addition.)

The elements studied have been listed in order in Table III, starting from the most beneficial and ending with the most detrimental, based on additions of equal per cent by weight. Also included in this table are comments on the appearance of the slag on the surface of the pins, coring, second phases, grain-boundary mobility, and incidence of internal cracks. With few exceptions, additions which produce a high susceptibility to surface hot-cracking are also the ones which, at higher concentrations, cause the occurrence of cracks at the interior of the pins. As-cast grain size does not seem to be a controlling factor in these small castings.

Even in these small pins as chill-cast into copper molds, it is frequently observed that the grain boundaries, which initially followed the irregular paths of the junctions between dendrites, have migrated appreciable distances. In Fig. 17, for example, it can be seen that the

TABLE III.—EFFECTS OF ALLOYING ELEMENTS ON HOT CRACKING AND STRUCTURE OF STAINLESS STEELS CONTAINING 16 PER CENT CHROMIUM AND 20 PER CENT NICKEL.

Alloying Element	Initial Slope, ΔM/Weight per cent (Surface Cracks)	Appearance of Slag on Surface of Cast Pin	Degree of Coring (Aqua Regia Etch)	Grain-Boundary Phases	Grain-Boundary Mobility	Internal Cracks	References
Nitrogen	2.3	dull dark gray to black (cratered surface)	faint	none	mobile	none	(**2,3,29**)
Molybdenum	0.50	light gray, granular	very strong	none	mobile	none	(**2–9,20,32**)
Manganese	0.32	reddish-black to black, glassy	faint	none	mobile	none	(**2,5,6,8,10–12,20,30,32**)
Tungsten	0.20	granular pink to pinkish brown	pronounced	none	mobile	none	(**8,12,13,32**)
Chromium	0.10	greenish-black to black	faint	none	mobile	none	(**3,11**)
Cobalt	0	dark brown to black	faint	none	mobile	none	(**7,12,32**)
Vanadium	0	dull, brownish black	moderate	none	mobile	none	(**5,6,12,32**)
Aluminum	0	granular, translucent pink to gray	faint	none	occasional sticking	none	(**32**)
Nickel	−0.10	greenish-black to black	faint	none	mobile	none	(**14–17**)
Tantalum	−0.50	black	faint	yes	occasional sticking	none	(**3,4**)
Copper	−0.90	reddish-black to black	faint	none	mobile	none	(**11,18,19,32**)
Silicon	−1.4	dark, transparent, glassy	faint	none	mobile	occasional	(**7–9,11,20–25,32**)
Columbium	−3.8	dull dark gray to black	moderate	scattered to continuous	stuck	severe	(**3,4,6,10–12,15,21,22,26,27,32**)
Titanium	−5.4	dark brownish, purple, glassy	faint	yes	partial sticking	none	(**7,15,28,32**)
Carbon	−6.7	dull greenish gray to black	faint	none	mobile	none	(**3,4,5,14,15,23–25,32**)
Hafnium	−19	thick gray scum	faint	yes	stuck	severe	
Zirconium	−35	dull dark gray	moderate	yes	stuck	severe	(**32**)
Boron	−300	pinkish gray, translucent, granular	faint	continuous	stuck	severe	(**12,31,32**)

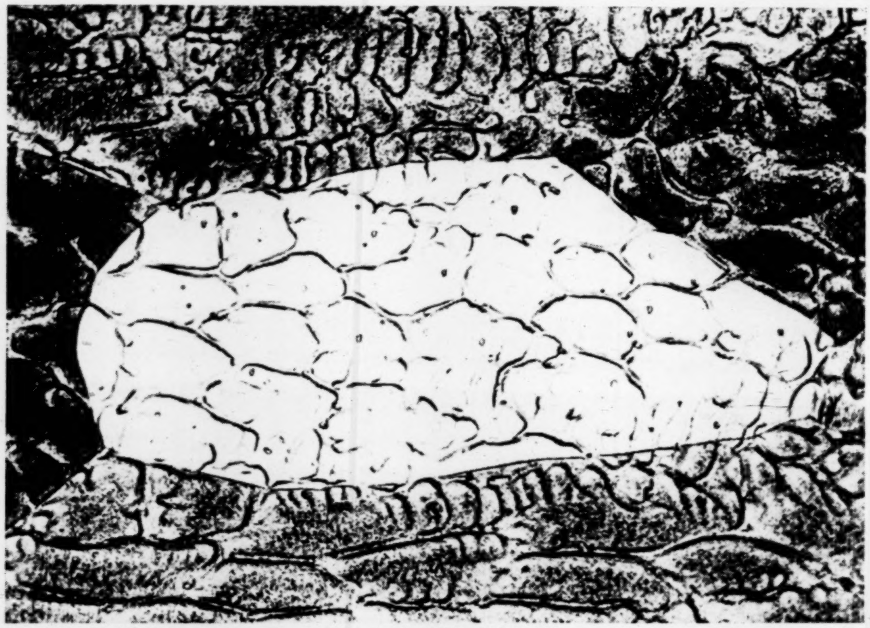


FIG. 17.—Mobile Grain Boundaries Ignore Solid-Solution Segregation (Coring) in Heat 7137 Steel (16 per cent Chromium, 20 per cent Nickel, 0.4 per cent Manganese, 1.25 per cent Silicon) (×500).

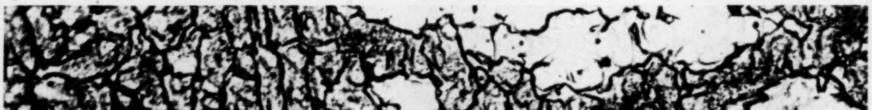


FIG. 18.—Grain Boundaries Are Stuck by the Phase Present at the Irregular Dendrite Boundaries in Heat 7052 Steel (17.2 per cent Chromium, 17.2 per cent Nickel, 1.59 per cent Manganese, 0.17 per cent Silicon, and 1.72 per cent Columbium) (×500).

grain boundaries in this steel are very smooth and are able to move through the alloy with no hindrance from the cored regions at the original cell and dendrite boundaries. A similar structure is found in the highly cored steel containing tungsten illustrated in Fig. 2. However, if coring or segregation is sufficient to lead to the appearance of a second phase at the dendrite and cell boundaries, the ability of the boundary to move is restricted and may eventually be fully impaired as the amount of the phase increases. This behavior is characteristic of alloys containing B, Zr, Hf, and Cb. The grain boundaries revealed by the contrast etch in Fig. 18 are pinned to the original dendrite boundaries by a relatively small amount of second phase. Also, at the lower magnifications in Fig. 3 and 4, the irregular and fixed positions of the boundaries are apparent. The possible significance of these observations is illustrated in Fig. 2 where a hot crack can be seen to have stopped at the point at which it encountered a migrating grain boundary. Table III shows that there is a clear correlation between the presence of second phases and restriction of grain-boundary motion and between both of these and the amount of hot cracking.

DISCUSSION

Comparison of CPT Test Results with Experience:

Before the results of these experiments are applied to the solution of weldability problems, it should be shown that the CPT test results correlate with hot cracking of welds. Although CPT test ratings are not yet available on many heats of steels for which weld-cracking studies have been made, indirect proof of such a correlation can be given.

The last column of Table III contains a listing of papers which refer to the effects of alloying elements on hot cracking of welds. In only a few of these references have composition variables been evaluated systematically by a test procedure that yields a quantitative result. Usually the general effects of the elements are deduced by comparing field or laboratory welding experience for a relatively limited number of commercial or experimental electrodes. In the complex stainless steels, which contain several additions, more information is needed to differentiate the effects of the individual elements.

The beneficial effects of Cr, Mn, N, and Mo and the detrimental effects of Ni, Si, Cu, Ti, Cb, Zr, and B on hot cracking, as determined by the CPT test results, are qualitatively supported by other investigators. The neutral effects of aluminum, cobalt, and vanadium have been previously reported by Gueussier and Castro (32). Although the data in the literature on the effect of tungsten are very limited, it can be concluded that this element is either beneficial or only mildly detrimental, because of its use in several successful electrodes and alloys; this effect is also consistent with the data in this paper. During the inert-gas welding of unrestrained, $\frac{5}{8}$-in. thick grooved plates of stainless steel containing columbium, zirconium or hafnium (with electrodes of the same composition as the base plate), extensive transverse and longitudinal hot cracking occurred in the weld bead. These same alloys had very poor ratings in the CPT test. Preliminary tests on experimental steels stabilized with tantalum have indicated that such steels are much less susceptible to hot cracking than if columbium is used. Although the majority of authors state that high carbon reduces hot cracking, the CPT test supports Medovar's finding (15) that low carbon is to be preferred for reduced hot cracking. For all of the 18 alloying elements there is some evidence or an indication that the CPT test measures the same hot-cracking tendencies that are important in weld-metal cracking.

Design of Alloys:

On the basis of the established quantitative effects of the various elements, it is now possible to design fully austenitic stainless steels that are resistant to hot cracking. In the base composition, chromium should be as high as possible without giving rise to delta ferrite and sigma; nickel should be as low as possible without introducing delta ferrite. Of the deoxidizers, manganese should be much higher than normally employed in chromium-nickel steels, aluminum is permissible in small amounts, and silicon should be low. Higher strength can be obtained by the addition of the solid-solution strengtheners vanadium, tungsten, and molybdenum, the latter two actually increasing resistance to cracking as well as improving strength. Carbon should be low, but nitrogen may be relatively high. If carbide stabilization is required or even greater strength desired, the preferred addition is tantalum, since both titanium and columbium drastically increase the susceptibility of steels to hot cracking.

The microstructure, as well as the composition, is an important variable in controlling hot cracking. When there is no objection to ferrite, the maximum possible resistance to hot cracking can be obtained by adjusting chromium and nickel (or the other ferrite- or austenite-forming elements in the alloy), to yield 7 to 10 per cent delta ferrite in the as-cast structure. If elements are added that lead to the occurrence of a small amount of a second phase (other than ferrite) at the dendrite boundaries, the grain boundary mobility is restricted with a resulting increase in susceptibility to hot cracking.

As is the case with any problem of alloy design, in addition to the specific object of avoiding cracking during casting or welding the metallurgist must also provide other characteristics such as high-temperature strength with long-time stability or corrosion resistance. The data presented are proving their value as guides in the development of stainless steels with improved resistance to hot cracking. Moreover, when the composition must be adjusted to obtain the optimum combination of the other desired properties required to fulfill various industrial applications, the CPT test provides a quick check on the cracking tendencies of these alloys.

CONCLUSIONS

1. Since the effects of alloying elements on hot cracking of stainless steels, as determined by the Cast Pin Tear test, agree with qualitative statements found in the literature based on miscellaneous test procedures and welding experience, it seems likely that the CPT test is capable of predicting the quantitative effects of alloying additions on the hot cracking of deposited weld metal.

2. The effects of 18 alloying elements on susceptibility to hot cracking of austenitic stainless steels in the CPT test have been evaluated quantitatively. Based on equal weight percentages, the elements rank in the following order (starting with the most beneficial and ending with the most detrimental): N, Mo, Mn, W, Cr, Co, V, Al, Ni, Ta, Cu, Si, Cb, Ti, C, Hf, Zr, and B.

3. Grain-boundary fractures that are produced at the surface of CPT test pins appear to be caused by hot cracking in the temperature range just below the solidus, although in alloys containing B, Cb, Hf, and Zr cracking may occur while there is still liquid present within the pin.

4. In alloyed stainless steels free of delta ferrite the appearance of a second phase at the dendrite and cell boundaries severely limits the mobility of grain boundaries; the combined effect of the phase and the restricted motion is a marked increase in susceptibility to hot cracking.

5. The data obtained provide a basis for the design of stainless steel electrodes of improved resistance to hot cracking during welding and of casting compositions resistant to hot tearing.

Acknowledgments:

The author is indebted to Mr. John P. Webb for his untiring efforts in the preparation of thousands of alloys and to other members of the Metallurgy Department at the Westinghouse Research Laboratories for metallographic work and helpful discussions.

REFERENCES

(1) F. C. Hull, "Cast-Pin Tear Test for Susceptibility to Hot Cracking," *Welding Journal*, Vol. 38 (4) (Research Supplement) pp. 176s–181s (1959).

(2) W. E. Ellis and M. Fleischmann, "The Substitution of Manganese for Nickel in 16-25-6," *Transactions*, Am. Soc. for Metals, Vol. 46, pp. 1039–1054 (1954).

(3) A. Hoerl and T. J. Moore, "The Welding of Type 347 Steels," *Welding Journal*, Vol. 36 (10) (Research Supplement), pp. 442s–448s (1957).

(4) R. D. Thomas, Jr. and L. K. Poole, "Mechanical Properties of Modified Type 347 Weld Metals," *Welding Journal*, Vol. 34, (Research Supplement), pp. 583s–595s (1955).

(5) W. L. Lutes and H. F. Reid, Jr., "Comparative Properties of Electrodes for Arc Welding Austenitic Manganese Steel," *Welding Journal*, Vol. 35, pp. 776-783 (1956).

(6) W. T. DeLong and H. F. Reid, Jr., "Properties of Austenitic Chromium-Manganese Stainless-Steel Weld Metal," *Welding Journal*, Vol. 36, (Research Supplement), pp. 41s–48s (1957).

(7) E. Kauhausen and H. A. Vogels, "Successful Manufacture and Use of All-Austenitic Welding Electrodes," *Metal Progress*, Vol. 67 (1), pp. 129–136 (1955).

(8) H. F. Tremlett, "The Problem of Embrittlement in the Welding of Austenitic Heat Resistant Steels," *British Welding Journal*, Vol. 4, pp. 283–287 (1957).

(9) "Stainless Steel Welding Rods and Bare Electrodes," *Welding Journal*, Vol. 33 (5), pp. 433–437 (1954).

(10) R. D. Thomas, Jr., "Crack Sensitivity of Chromium-Nickel Stainless Steel Weld Metal," *Metal Progress*, Vol. 50, No. 3, pp. 474–479 (1946).

(11) A. J. Williams, P. J. Rieppel, and C. B. Voldrich, "Literature Survey on Weld-Metal Cracking," *WADC Technical Report*, No. 52–143, Wright Air Development Center; *Abstract in Metal Progress*, Vol. 67, No. 2, p. 146, No. 3, p. 164 (1955).

(12) H. Buchholtz, W. Ruttmann, and R. Schinn, "Metallic Materials for a Steam Power Plant Operating at 1130 F," *Metal Progress*, Vol. 65, No. 1, pp. 116–120, 180, 182, 184, 186 (1954).

(13) B. A. Movchan and L. A. Poznyak, "On the Nature of Intergranular Cracking of Welds at High Temperatures," *Avtomaticheskaya Svarka*, Vol. 8 (6), pp. 59–72 (1954). (H. Brutcher, Translation 3572.)

(14) G. E. Linnert, "Welding Type 347 Stainless Steel Piping and Tubing," *Welding Research Council Bulletin*, No. 43, Oct. 1958.

(15) B. I. Medovar, "On the Nature of Weld Hot Cracking," *Avtomaticheskaya Svarka*, Vol. 7 (4), pp. 12–28 (1954). (H. Brutcher, Translation 3400.)

(16) F. M. Beckett and R. Franks, "Effects of Columbium in Chromium-Nickel Steels," *Transactions*, Am. Inst. Mining, Metallurgical, and Petroleum Engrs., Vol. 113, pp. 143–162 (1934).

(17) W. O. Binder, "Interchangeability of Cb and Ta in Type 347 Stainless," *Metal Progress*, Vol. 59, No. 2, pp. 219–227 (1951).

(18) G. E. Linnert, "Welding Precipitation Hardening Stainless Steels," *Welding Journal*, Vol. 36 (1), pp. 9–27 (1957).

(19) R. Wentworth, "Copper in Type 347 Weld Metal," *Welding Journal*, Vol. 32

(7) (Research Supplement), pp. 347s–351s (1953).

(20) S. A. Herres and A. M. Turkalo, "Welding of Hardenable Steels with High-Alloy (Austenitic) Electrodes," *Welding Journal*, Vol. 25 (10) (Research Supplement), pp. 669s–696s (1946).

(21) T. E. Kihlgren and C. E. Lacy, "The Control of Weld Hot Cracking in Nickel-Chromium-Iron Alloys," *Welding Journal*, Vol. 25 (11) (Research Supplement), pp. 769s–775s (1946).

(22) E. Bishop and W. H. Bailey, "Weld Metal Properties and Welding Characteristics of Two Austenitic Steels Used for Gas Turbine Rotors," Symposium on High-Temperature Steels and Alloys for Gas Turbines, *Special Report No. 43*, The Iron and Steel Inst., London (England), July 1952.

(23) L. K. Poole, "The Incidence of Cracking in Welding Type 347 Steels," *Welding Journal*, Vol. 32 (8) (Research Supplement), pp. 403s–412s (1953).

(24) R. M. Curran and A. W. Rankin, "Welding Type 347 Stainless Steel for 1100 F Turbine Operation," *Welding Journal*, Vol. 34 (3), pp. 205–213 (1955).

(25) O. R. Carpenter and N. C. Jessen, "Some Factors Controlling the Ductility of 25 per cent Chromium-20 per cent Nickel Weld Deposits," *Welding Journal*, Vol. 26 (12) (Research Supplement), pp. 727s–740s (1947).

(26) H. M. Soldan and C. R. Mayne, "Cracking Tendency in Heavy High-Temperature Steam Piping," *Welding Journal*, Vol. 36 (3) (Research Supplement), pp. 141s–150s (1957).

(27) P. P. Puzak and H. Rischall, "Further Studies on Stainless Steel Hot Cracking," *Welding Journal*, Vol. 36 (2) (Research Supplement), pp. 57s–61s (1957).

(28) J. J. Vagi and D. C. Martin, "Welding of High-Strength Stainless Steels for Elevated-Temperature Use," *Welding Journal*, Vol. 35 (3) (Research Supplement), pp. 137s–144s (1956).

(29) R. A. Perkins and W. O. Binder, "Resistance to Hot-Cracking of Chromium-Nickel Steel Welds," U. S. Patent No. 2,871,118, Jan. 27, 1959.

(30) G. E. Linnert and R. M. Larrimore, "Stainless Steel for Weld," U. S. Patent No. 2,894,833, July 14, 1959.

(31) G. E. Claussen, "Boron Causes Hot Cracking in Submerged-Arc Welds," *Welding Journal*, Vol. 38 (4), p. 300 (1959).

(32) André Gueussier and René Castro, "Experimental Study of Solidification Cracking in Steels—Influence of Impurities," *Revue de Métallurgie*, Vol. 57, No. 2, pp. 117–134 (1960).

DISCUSSION

MR. HENRY M. SOLDAN[1] (*presented in written form*).—The author is to be congratulated on the very interesting investigation which he has made concerning the effects of alloying additions to austenitic chromium-nickel steels with respect to hot cracking. The data presented bear out the practical conclusions which we had reached concerning the detrimental effects of some alloying elements, particularly columbium and titanium. In our work these have for the most part proved to be detrimental with respect to hot cracking in heavy sections.

It is unfortunate, as we see it, that the author did not concentrate his work around the existing commonly used alloys of the 300 series, rather than make his variations around a 16-20 alloy with which many of us in the utilities field have had no practical experience. We hope that in his closure he will include some data on types 321 and 347 steels and variations thereof. Variations in the percentage of columbium or titanium additions to the 18-8 type 304 alloy with respect to hot cracking, if available, would furnish some very useful information.

The author suggests that where there is no objection to ferrite, hot cracking can be minimized by adjusting the elements to yield 7 to 10 per cent delta ferrite in the as-cast structure. Some 10 years ago, we found that some of our weldments had become extremely brittle and had suffered a severe loss in impact strength, particularly 19/9 W-Mo[2] after a relatively short time at 1050 F. As a result, a consulting group, of which Public Service Electric and Gas Co. formed a part, decided to develop electrodes which would deposit a maximum of 3 to 5 per cent ferrite in the finished weld, to reduce the amount of sigma and the tendency to embrittle.

---

[1] Public Service Electric and Gas Co., Newark, N. J.

A fact not to be lost sight of is that in heavy wall sections, for high-temperature service, parent metal rather than weld metal cracking has been the major difficulty. This led to the development of the 16-8-2 electrode which has been successfully employed and takes advantage of its inherent ductility to reduce the stresses in the adjacent parent metal weld-heat-affected zone upon cooling. It has been necessary, in order to make some field repairs in base metal having a hot cracking tendency, to use electrodes which deposit an extremely ductile weld metal.

In the author's closure we would also like to see some reference made to "Kromarc 55" steel which we understand is about to be advocated as a compatriot of 16-8-2.

MR. J. J. HEGER.[3]—Does the relationship found between chemical composition and weld metal cracking apply to the cracking that occurs in the heat-affected zone of welds?

MR. S. J. ROSENBERG.[4]—Has the author done or thought of doing any work relating his cast pin tear test to the hot workability of these materials? I ask this question because we recently melted an austenitic stainless steel containing 4 per cent copper and it proved to be quite hot short. The author's Fig. 10 shows that copper, at least up to 2 per cent, increases the susceptibility to hot cracking and thus appears to corroborate our single experience. It is possible that a correlation exists between the author's relatively simple test and the hot workability of steels and it may be advisable to explore this possibility.

In view of the tremendous amount of interest in vacuum-melting, it is rather disappointing to note the generally inconclusive effect of vacuum melting upon the susceptibility to hot cracking. The data presented by the author indicate that vacuum melting may increase, decrease, or have no effect upon the tendency toward hot cracking.

MR. A. B. WILDER.[5]—The author has developed a cracking susceptibility test (CPT) for austenitic stainless steels. Test results indicate that certain steels, such as types 321 and 347, are unsuitable, yet large quantities of these steels have been used successfully. Does the author have any information that relates the CPT test to field welding conditions, hot-mill rolling characteristics, or any other conditions which are encountered in the production and use of these alloys?

Also it will be of interest to know if the conditions encountered in alloys susceptible to cracking in the cracking susceptibility test (CPT) may be detected by X-ray or other nondestructive tests in actual weldments.

MR. F. C. HULL (*author's closure*).—The author is glad to have the additional confirmation by Mr. Soldan of the detrimental effects of titanium and columbium on hot cracking in the welding of heavy sections in practice. Laboratory tests are frequently used for screening purposes in alloy development, but the real criterion of the value of a test is how well it can predict performance in service.

---

[2] 19/9 W-Mo is a particular name for an alloy containing 19 per cent chromium, 9 per cent nickel, with tungsten, molybdenum, columbium, and titanium.

[3] Applied Research, U. S. Steel Corp., Pittsburgh, Pa.

[4] Metallurgist, National Bureau of Standards, Washington, D. C.

[5] Chief Metallurgist, National Tube Division, U. S. Steel Corp., Pittsburgh, Pa.

The selection of the 16 per cent chromium, 20 per cent nickel base for the present study was made after a careful study by several people at the Westinghouse Research Laboratories. It was recognized that this composition differs markedly from the 300 series alloys and that, therefore, the application of the data to practice might not be direct. This consideration was overweighed by our desire to be able to evaluate the effects of large additions of 18 elements in a single base composition without having the results obscured by the influence of variable amounts of delta ferrite. Many of the 300 series steels may contain some delta ferrite as cast, depending on their actual composition within the limits of the current specifications. It would clearly not have been possible to study the effects of 4 to 5 per cent additions of chromium, tungsten, molybdenum, or vanadium except in such an alloy as the one selected. Even so, heat VM419 with 4.6 per cent vanadium contained 1.8 per cent delta ferrite.

To use one base composition for the study of these four elements and a different base for another group would have made it difficult to make quantitative comparisons of the effects of elements such as manganese, molybdenum, columbium, and titanium. Figures 8 and 9 of the paper show that the effects of manganese and silicon are similar in alloys of widely different chromium and nickel contents. It is therefore believed that the composition effects reported would apply in the range of the 300 series stainless steels as long as the complicating effects of composition on delta ferrite formation are avoided.

One of the objections to delta ferrite in weld deposits, to which I referred in the paper, is its tendency to embrittle the welds by transformation to sigma phase in high-temperature service. Although this behavior may be fairly well known, since it was not specifically mentioned, it is a valuable addition to the paper to have Mr. Soldan's warning on the effects of ferrite and his remarks on the development of low ferrite electrodes. In fact, one of the original goals of the investigation of which this paper is a part was to find a fully austenitic steel so resistant to hot cracking that it would not need delta ferrite in its weld deposits to prevent microfissuring. This goal has been attained, as will be described later.

Both Mr. Soldan and Mr. Heger have referred to the importance of base-metal cracking which occurs in the heat-affected zone of a weld. The writer and Mr. C. S. Williams, also from the Westinghouse Research Laboratories, have examined the effects of composition on wrought base metal heat-affected zone behavior as evaluated by a modified Rensselaer Polytechnic Inst. hot ductility test and hot cracking susceptibility of cast structures as determined by the cracking susceptibility test (CPT). In most cases there is good correlation between the results of the two tests. An unexpected dividend of the present paper is thus the possibility of developing weldable base-metal compositions from the CPT test results as well as compositions that are resistant to hot cracking for weld deposits and castings.

Mr. Rosenberg has asked if the CPT test can predict the hot workability of stainless steels. This interesting possibility has not been explored. In the case of compositions containing delta ferrite as cast, the CPT test could definitely not be applied, for the test would predict a low susceptibility to hot cracking, whereas production hot-rolling experience shows such compositions to be highly prone to cracking.

The experiments in this paper were aimed at determining the effects on hot cracking of major amounts of alloying elements. Thus the differences in gas content or minor impurities, some of which may be beneficial and others detrimental, produced by air or vacuum melting were not investigated. Fortunately for stainless steel users, it appears that major improvements in susceptibility to hot cracking can be obtained by inexpensive variations in composition of some of the common alloying elements rather than requiring superpurity charge materials and exotic melting techniques.

In answer to Mr. Wilder's remark on the successful use of types 321 and 347 steels, I would like to emphasize that this paper was aimed initially at hot cracking of only fully austenitic cast alloys. In the CPT test, titanium and columbium produce a high susceptibility to hot cracking. All of the commercial stainless steel welding electrodes that I am aware of that contain titanium or columbium require delta ferrite in the as-deposited weld metal to prevent hot cracking in restrained joints. Since delta ferrite is detrimental in high-temperature applications, as Mr. Soldan has described, ferrite is used, obviously not from choice, but because the consequences are less objectionable than cracked welds.

From the correlation mentioned previously, I would expect types 321 and 347 base metals to be more susceptible to heat-affected zone cracking during welding than types 304 and 316. This does not mean that types 321 and 347 cannot be used successfully, for they have been, as Mr. Wilder states; but it indicates that preheat, size of parts, rigidity of the joints, welding variables, and impurity levels would have to be controlled more closely to obtain crack-free welds. A comparison of the field welding and service experience on types 316 and 347 stainless steel piping for steam power stations confirms that type 347 is subject to many more difficulties. I have found that alloys with mold numbers in excess of about 13 for a cracking index of 40 are only marginally acceptable for production welding of heavy-walled restrained joints. Types 321 and 347 steels unfortunately occasionally fall in this range.

In alloys of high CPT test susceptibility ratings, longitudinal and transverse weld bead cracks that were visible to the unaided eye occurred in welds in $\frac{5}{8}$ in. thick unrestrained plates. In other cases, cracks that were only found by examination with a microscope could have escaped detection by X-ray or dye penetrant inspection. The most satisfactory solution to the weld cracking problem is to design base- and weld-metal compositions so that there is a sufficiently wide margin of safety to compensate for all the uncontrolled processing and compositional variables that can enter into the complex process of fusion welding.

Through the application of the design principles given in this paper, the Westinghouse Electric Corp. has developed a stainless steel of exceptionally high resistance to hot cracking. This alloy, "Kromarc 55," about which Mr. Soldan inquired, has a nominal composition of 16 per cent chromium, 20 per cent nickel, 9.5 per cent manganese, 0.3 per cent silicon, 2.25 per cent molybdenum, 0.04 per cent carbon and balance iron, and it has been produced as forgings, plate, castings, and welding electrodes. Steam piping of type 316 steel and "Kromarc 55" steel nozzle chamber castings have been welded in massive restrained joints with a fully austenitic weld deposit of this same composition without weld metal or heat-affected zone cracking. Since delta ferrite is not needed to prevent weld-bead hot cracking, the welded joints do not require solution treatment, as has been the practice in many steam plant installations. "Kromarc 55" steel has about the same high-temperature strength but has considerably less susceptibility to hot cracking than type 316 stainless steel which is considered to have excellent weldability. Thus the prospects appear favorable for the development of alloys of even higher strength for welded applications.

CARBON:STRENGTH RELATIONSHIPS IN 5 PER CENT CHROMIUM ULTRA-HIGH-STRENGTH STEELS*

By John C. Hamaker, Jr.[1] and Eugene J. Vater[1]

Synopsis

The effects of carbon content and vacuum melting on the mechanical properties of 5.0 per cent chromium, 1.3 per cent molybdenum, 0.5 per cent vanadium ultra-high-strength steel were investigated between −320 and +1100 F. All compositions exhibited the soft annealed properties, deep air hardenability, high M_s temperatures, and secondary hardening on tempering characteristic of the 0.40 per cent carbon production steel used for several years in the 220,000 to 300,000 psi ultimate strength range.

Vacuum melting resulted in marked improvements in ductility and toughness, surpassing variations in both carbon content and heat-treated strength level in effectiveness. Two potentially useful modifications of the commercial 0.40 per cent carbon steel were studied: a 0.20 per cent carbon grade capable of 260,000 psi ultimate strength with high ductility, hot strength, and potentially good weldability; and a 0.50 per cent carbon grade capable of 330,000 psi ultimate strength, 275,000 psi yield strength, with high fatigue and impact strength.

The importance of secondary hardening for the removal of retained austenite and residual stress, with resulting improvements in fatigue, notch, and transverse ductility properties, is discussed in the light of present trends to higher strength levels.

The medium alloy steel system containing 5.0 per cent chromium, 1.3 per cent molybdenum, and 0.5 per cent vanadium offers an interesting and strategically important series of alloys for study in the field of ultra-high-strength materials for 200,000 to 330,000 psi service. With carbon contents in the normal 0.20 to 0.50 per cent range for structural steels, the system exhibits heat-treating characteristics that are unique to the structural field.

Extremely deep martensitic hardenability in air, through sections up to 20 in. cube, minimizes distortion, danger of cracking, and residual stress. Relatively high M_s temperatures, in the 500 to 700 F range, result in low retained austenite contents that can be readily transformed by tempering.

Of greatest importance is the secondary hardening and accompanying retained austenite removal, on tempering at 950 to 1000 F. As described by Crafts and

---

* Presented at the Sixty-third Annual Meeting of the Society, Atlantic City, N. J. June 26–July 1, 1960.

[1] Director of Research and Metallurgical Engineering and Research Metallurgist, respectively, Vanadium-Alloys Steel Co., Latrobe, Pa.

Lamont (**1**)[2] and Kuo (**2**,**3**), secondary hardening involves the precipitation of fine carbides from martensite, similar to the tempering of low alloy steel. However, in secondary hardening steels, these are alloy carbides of the Mo_2C and VC type which precipitate as extremely fine particles on heating into the 900 to 1100 F range, providing a hardness level approximately equivalent to that in the untempered steel, and accompanied by exceptional strength.

Cohen and his associates (**4**) showed that the high tempering temperatures permitted by secondary hardening "condition" the retained austenite so that its M_s temperature is effectively raised, and it subsequently transforms to martensite on cooling to room temperature. A second tempering treatment then toughens the newly formed martensite to obtain a fully tempered martensite structure. In heavier sections, where larger amounts of austenite tend to be retained in all steels due to cooling rate stabilization, several tempering operations are necessary to complete the transformation of the austenite to martensite and subsequently temper the latter. Each temper raises the M_s temperature of the remaining austenite successively higher to permit further transformation. Ruff (**5**) points out that multiple tempering effects complete transformation only with moderate amounts of austenite resulting from relatively high M_s temperatures in the base composition. Laboratories using X-ray diffraction equipment capable of detecting 0.5 to 1.0 per cent retained austenite have reported that the 0.40 per cent carbon steel in this system is free of austenite after triple tempering to the 290,000 psi ultimate tensile level or double tempering to the 260,000 psi tensile level.[3]

Retained austenite is becoming an increasingly significant factor in low alloy steels as higher strength levels are employed for structural purposes. At the 180,000 psi tensile level, which until a few years ago was accepted as maximum for aircraft purposes, austenite retained during the quenching operation was quite effectively transformed to bainite by the relatively high tempering temperatures employed. However, as strength levels of 260,000 to 300,000 psi are sought by the use of lower tempering temperatures on low alloy steels, the retained austenite resists transformation by either long tempering treatments or repeated heating to the tempering temperature. Various refrigeration and baking cycles are used to minimize the austenite and stabilize it as much as possible against further transformation to brittle martensite in service. These treatments are quite effective in stabilizing the austenite against thermal transformation under, for example, arctic conditions, but may not fully prevent stress-induced transformation caused by the normal operating stresses encountered in service. Averbach, Lorris, and Cohen (**7**) obtained complete transformation of retained austenite to fresh martensite in AISI 2340 tension bars before the onset of neckdown, and found transformation in Charpy V-notch impact specimens at distances up to 0.040 in. from the fracture.

Vajda, Hauser, and Wells (**8**) showed that silicon additions of 1.0 and 1.5 per cent to AISI 4340 and 8740 steels, similar to those being used to raise strength capabilities and avoid the 500 F embrittlement range, can produce up to 25 per cent retained austenite in heavy sections. They further observed that the tempering temperatures necessary to transform this austenite to bainite increased progressively with the silicon content from 600 F for the base 4340 analysis to 700 F for 1.0 per cent silicon and 800 F for 1.5 per cent silicon. Thus, the higher tempering temperature advantages of the silicon modified low alloy steels may be offset by greater quantities of austenite retained at these tempering temperatures.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

[3] Private communication North American Aviation, Inc., and Douglas Aircraft Co.

Frankel, Bennett, and Pennington (**9**) concluded that up to 10 per cent retained austenite lowers the fatigue strength of steels appreciably, due to its stress-induced transformation to untempered martensite. Recent reports of "missile pressure vessel fatigue," in which failures occurred at low stresses after initially meeting specifications on proof tests, might conceivably be a related phenomenon. Retained austenite could transform to fresh martensite on the first stressing, followed by embrittlement from this untempered martensite on the second pressure application.

With the present trends toward more heavily alloyed low alloy steels to improve hardenability and strengthening capacity, together with the heavier sections being employed in large aircraft structures, it is evident that this retained austenite problem may assume increasing significance in nonsecondary hardening steels. Other advantages of secondary hardening are readily apparent: retention of high-strength properties to elevated temperatures, elimination of the danger of strength loss due to overheating in service, and high-temperature heating for residual stress or hydrogen removal after plating or processing.

Preliminary alloy development research on variations from the 5.0 per cent chromium, 1.3 per cent molybdenum, 0.5 per cent vanadium system indicated that lower alloy contents fail to retain the full secondary hardening tendency (that is, the secondary hardness peak fails to achieve the untempered hardness level), whereas higher alloy contents or additions of new alloying elements either decrease ductility or result in rapid increases in retained austenite content that resist transformation even with multiple tempering. The 0.40 per cent carbon steel in this system,[4] introduced for ultra-high-strength structural purposes several years ago (**10**), was rapidly adopted for aircraft and missile applications and has been furnished to guaranteed minimum specifications since June 1957.[5] Ruff, in a recent series of articles in *Metal Progress* (**5,6**), discussed the evaluation and fabrication of this steel for critically stressed airframe structures in a multi-Mach production aircraft. Further applications on welded missile pressure vessels, high-strength threaded fasteners, aircraft skins and structures, catapult hooks, shafts, and high-speed rotors have also been described (**11–15**). Forging, welding, deep drawing, machining, and other fabricating operations are generally reported equivalent to low alloy steels of similar strength capability (**5,6,15–17**).

During preliminary research on this steel, it was noted that higher and lower carbon modifications provided special combinations of properties that might augment those available in the 0.40 per cent carbon grade. Applications have since arisen indicating potential requirements for these modifications. Among these, on the low carbon side, are improved weldability in the hardened condition, and higher ductility at strength levels below the 260,000 to 300,000 psi optimum range of the 0.40 per cent carbon steel; while a higher carbon modification might be used for still higher strength potentials, where design criteria can be adjusted for the accompanying lower ductility levels.

Accordingly, a more extensive program was undertaken to establish the effect of carbon content between 0.20 and 0.50 per cent on the room temperature tensile, sheet tensile, impact, and fatigue properties, impact transition behavior, and elevated temperature tensile and impact properties, of 5 per cent chromium steels. From this study, the properties of higher and lower carbon modifications could be compared with those of the 0.40 per cent carbon production steel for which property:performance relationships have already been established.

---

[4] Frequently designated "Vascojet 1000."

[5] MB 1000 (for bars and billets) and MS 1000 (for sheet and plate), guaranteed minimum specifications, Vanadium-Alloys Steel Co.

TABLE I.—COMPOSITIONS OF STEELS INVESTIGATED, PER CENT.

Steel	Melt	Carbon	Chromium	Molybdenum	Vanadium	Silicon	Manganese	Sulfur	Phosphorus
X2	air	0.22	5.17	1.21	0.52	0.82	0.27	0.026	0.014
X3	air	0.305	5.03	1.30	0.50	0.86	0.29	0.021	0.018
VJ	air	0.40	5.21	1.30	0.54	0.98	0.37	0.011	0.017
VJ-VM	vacuum	0.36	5.02	1.24	0.48	0.78	0.28	0.013	0.006
X5	air	0.48	4.83	1.35	0.49	0.86	0.25	0.021	0.014

TABLE II.—HEAT TREATING CHARACTERISTICS OF 5 PER CENT CHROMIUM STEELS.

Steel	Carbon Content, per cent	Austenitizing Temperature, deg Fahr	Fracture Grain Size Number	Rockwell Hardness, C Scale		Martensite Start Temperature, M_s, deg Fahr	Retained Austenite, per cent	
				As Air Cooled	Triple Tempered at 950 F		As Air Cooled[a]	After Multiple Tempering Above 990 F[b]
X2	0.22	1825 ± 25	6½	49.0	51.6	690	2.4	<1
X3	0.31	1850 ± 50	8	55.0	55.1	650	3.1	<1
VJ	0.40	1850 ± 50	9	57.5	56.5	520	6.9	<1
VJ-VM	0.36	1850 ± 50	8½	57.1	56.0	575	4.9	<1
X5	0.48	1850 ± 50	9½	61.6	58.0	480	8.7	...

[a] Calculated from (**18**).

[b] Based on footnote 3.

EXPERIMENTAL PROCEDURE

The compositions investigated are presented in Table I. Four electric furnace air-melted heats, melted to tool steel and aircraft quality standards, represented nominal carbon contents of 0.20, 0.30, 0.40 (the commercial steel), and 0.50 per cent. An additional induction vacuum-melted heat was included to study the effect of vacuum melting on properties in the 0.40 per cent carbon range. All heats were cast into 7- to 10-in. ingots, and forged and rolled into ½-in. square, ⅝-in. round, and ⅞-in. round bars, and 0.125 in. thick by 24 in. wide sheet, using tool steel manufacturing procedures. A final annealing operation, consisting of 3 hr at 1600 F, cooling 40 F per hr to 1000 F, then air cooling, gave the following range of annealed properties for all five steels:

ANNEALED PROPERTIES

Brinell hardness	160 to 200
Proportional limit, psi	20 000 to 25 000
0.2 per cent offset yield strength, psi	35 000 to 50 000
Ultimate tensile strength, psi	85 000 to 100 000
Elongation in 2 in., per cent	23 to 28
Reduction of area, per cent	60 to 70

Preliminary Heat-Treatment Studies:

Preliminary heat-treating studies were conducted to determine the optimum austenitizing temperature range, grain size, M_s temperature, retained austenite, and tempering characteristics of each steel. The results are summarized in Table II.

All steels except X2 showed best response to heat treatment from an austenitizing cycle of 30 min at 1850 ± 50 F, resulting in fine fracture grain sizes of Nos. 8 to 9½, corresponding to ASTM Nos. 7½ to 9.[6] Their grain coarsening temperatures of 2000 to 2050 F, approximately 200 F above the normal austenitizing temperature, indicated a factor of safety for hardening and finish forging operations. The 0.20 per cent carbon X2 steel coarsened slightly to a No. 5 fracture grain size at 1850 F, but retained No. 4½ grain size to 2100 F. An austenitizing cycle of 30 min at 1825 F was selected for this steel.

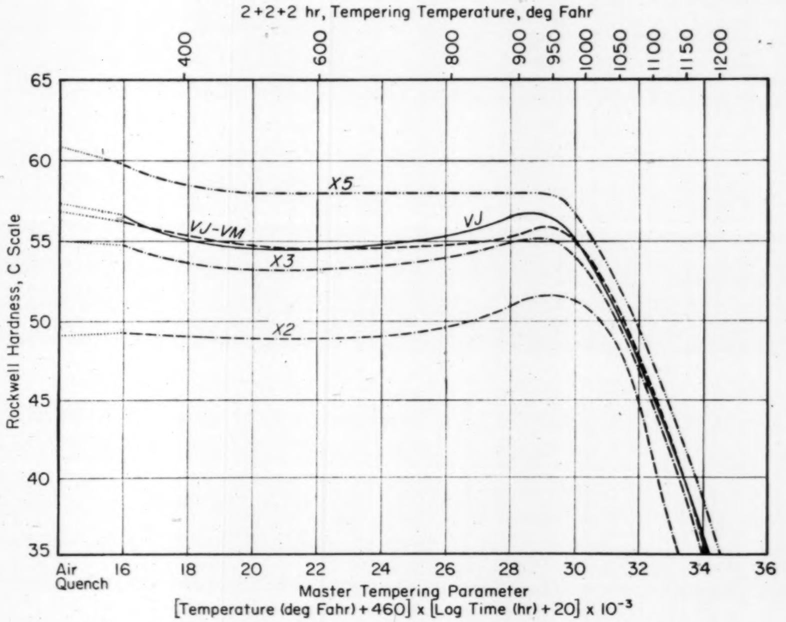


FIG. 1.—Master Tempering Curves for 5 per cent Chromium Steels.

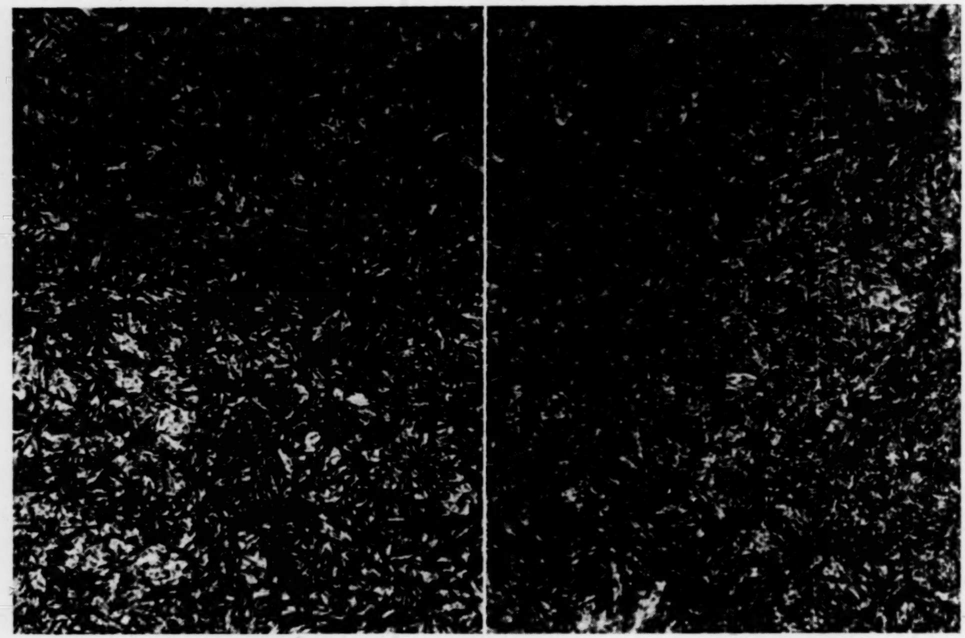
Parameter equation permits calculation of tempering temperatures and times for desired hardness (and strength) level, and safe operating temperatures and times for stable retention of these properties.

Due to the deep air-hardening characteristic of these steels, the hardness as cooled from the austenitizing temperature may vary somewhat with section size and is of secondary importance. Of greater significance is the hardness after multiple tempering to the secondary hardness peak and beyond, which provides uniform hardness of a given composition from heat to heat and with widely varying section size. The maximum secondary hardness for each steel is listed under the column "Triple Tempered at 950 F."

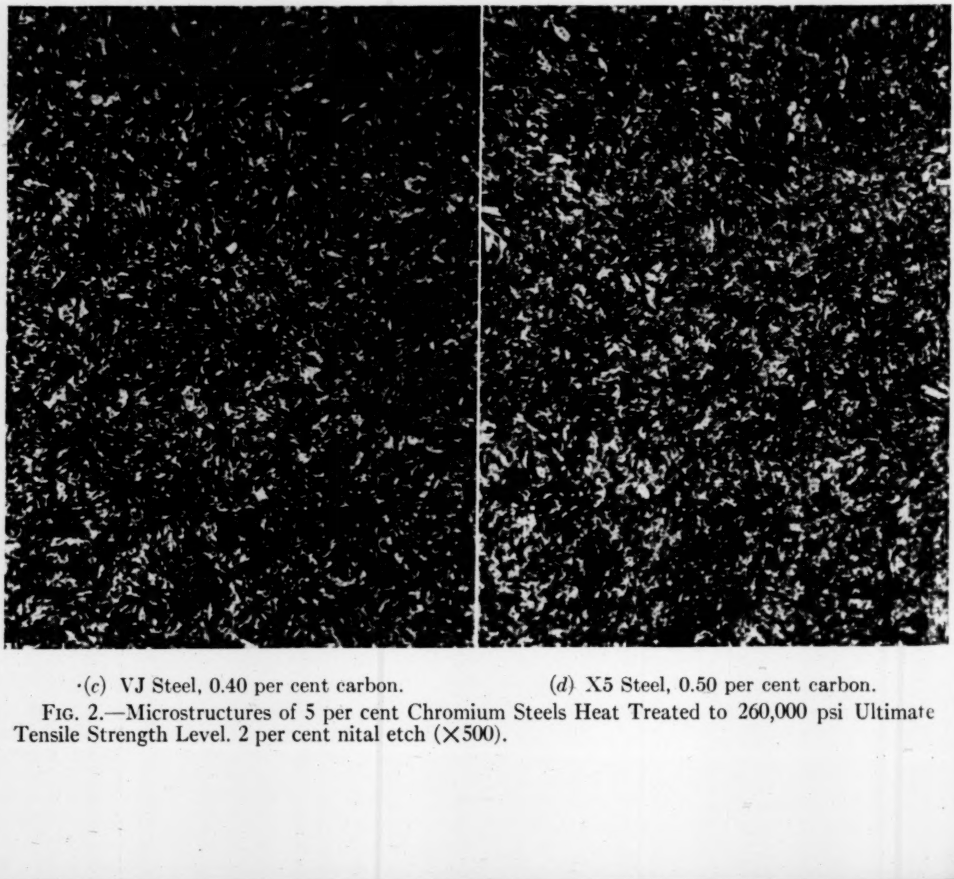
M_s temperatures, determined within

---

[6] Tentative Methods for Estimating the Average Grain Size of Metals (E 112 – 58 T), 1958 Book of ASTM Standards, Part 3, p. 506.



(*a*) X2 Steel, 0.20 per cent carbon. (*b*) X3 Steel, 0.30 per cent carbon.



·(*c*) VJ Steel, 0.40 per cent carbon. (*d*) X5 Steel, 0.50 per cent carbon.

FIG. 2.—Microstructures of 5 per cent Chromium Steels Heat Treated to 260,000 psi Ultimate Tensile Strength Level. 2 per cent nital etch (×500).

25 F by the conventional Greninger-Troiano quench-and-temper technique, tended to be higher than those for many low alloy steels, due to freedom from M_s-depressing alloys in the base composition. With the secondary hardening effect, multiple tempering results in essentially complete transformation to tempered martensite in the 0.20 to 0.40 per cent carbon steels. Although the retained austenite content of the tempered 0.48 per cent carbon X5 steel has not been experimentally determined, the high M_s and low as-cooled retained austenite would suggest essentially complete transformation with repeated tempers.

Master tempering curves for the five steels, using the conventional Holloman-Jaffe parameter (**19**), are illustrated in Fig. 1. Corresponding tempering temperatures for a triple 2-hr treatment are shown at the top of the graph. Both air-quenched hardness and tempered hardness consistently increase with carbon content in about the expected manner. However, the low carbon X2 steel exhibits a more pronounced tendency for secondary hardening than any of the others, despite its higher M_s temperature and very low retained austenite in the hardened condition. The high carbon X5 steel, on the other hand, possesses somewhat lower secondary hardness than would be expected from its as-cooled hardness level. This effect is probably related to the alloy-carbon ratio discussed by Kuo (**2**), in which he points out that greater secondary hardening effect is obtained from the more highly alloyed carbides obtained with lower carbon and high alloy contents. Furthermore, the fact that the 0.20 per cent carbon steel, with the highest M_s and least retained austenite, exhibited the greatest secondary hardening, supports the evidence that secondary hardening of these steels is primarily due to alloy carbide precipitation from tempered martensite rather than to retained austenite transformation.

Photomicrographs of the steels representing the four carbon contents, when heat treated to the 260,000 psi tensile level, are shown in Fig. 2. Uniform tempered martensite structures were obtained throughout, with no evidence of undissolved carbides or ferrite in any steel. For this strength range, the 0.20 per cent carbon X2 was slightly undertempered, resulting in a somewhat lighter etching structure than obtained in the other materials.

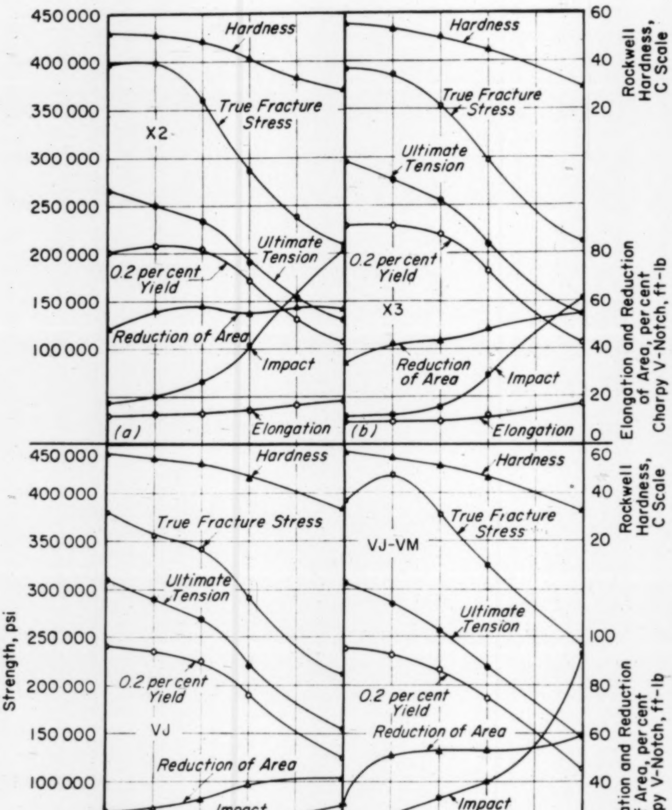
Specimen Preparation and Testing:

Heat treatment of all specimens was accomplished in neutral salt baths. Steels X3, VJ, VJ-VM, and X5 were preheated 15 min at 1125 F, austenitized at 1850 ±50 F for 30 min at temperature, and air cooled. Steel X2 was austenitized at 1825 F with an otherwise identical procedure. The specimens were then tempered in molten salt controlled to ±3 F at various temperatures between 950 and 1200 F. Triple tempering was employed throughout, each cycle consisting of 2 hr at temperature, with intermediate air cooling to room temperature.

For room and elevated temperature tension testing, triplicate 0.505 in. round specimens were prepared to ASTM E 8 - 57 T standard dimensions[7] from $\frac{7}{8}$ in.-diameter annealed hot-rolled bars. The 0.030-in. grinding stock was removed after heat treatment, followed by hand grinding with No. 100 emery paper to leave all surface marks at 45 deg to the axis. Sheet tension specimens were machined to ASTM E 8 - 57 T standard dimensions from 0.125 in. thick by 24 by 72 in. stock. Two groups of triplicate specimens were prepared for each steel, one set having their longitudinal axes

---

[7] Tentative Methods of Tension Testing of Metallic Materials (E 8 - 57 T), 1958 Book of ASTM Standards, Part 3, p. 103.



(a) 0.20 per cent carbon
(b) 0.30 per cent carbon
(c) 0.40 per cent carbon
(d) 0.40 per cent carbon vacuum melt.
(e) 0.50 per cent carbon

FIG. 3.—Range of Tensile and Impact Properties Obtained in 5 per cent Chromium Steels by Air Hardening from 1825 to 1850 F and Triple Tempering at Indicated Temperatures.

oriented parallel to and the other set transverse to the principal rolling direction of the sheet. After heat treatment, 0.012 in. was ground longitudinally from each of the flat sides, and the grip portions were softened to Rockwell hardness C 45 in a high frequency induction coil to facilitate gripping.

Tension testing was conducted on a Baldwin-Southwark 120,000-lb capacity tension machine, at a load application of 6000 lb, per min for the bar specimens and 1200 lb per min for the sheet. Autographic stress-strain curves were recorded for each specimen, using a microformer-type extensometer to the 0.2 per cent offset yield point, and a deflectometer to complete the record to fracture. Continuous measurement of specimen diameter during neckdown provided data for true stress - true strain curves. Elevated temperature tension tests at 800 to 1100 F were similarly conducted, using a multiple tap resistance furnace to provide temperature control of ±3 F during the test and ±5 F along the gage length in accordance with ASTM Recommended Practice E 21 – 58 T.[8]

V-notch Charpy impact specimens, following ASTM Methods E 23 – 56 T,[9] were prepared from $\frac{1}{2}$-in. square bar stock. To insure proper radius and freedom from transverse tool marks, the notches were milled prior to heat treatment, then circular ground at their bases after hardening, using an abrasive slurry on a wire 0.020 in. diameter rotated in a drill press. Such precautions are recommended in testing materials at hardness levels over Rockwell hardness C 45 (200,000 psi tensile strength) to insure that results reflect the radius of the notch rather than the radius of a scratch or tool mark at its base. Notch preparation was controlled by binocular microscope inspection, while the neutrality of heat treatment and adequacy of stock removal were checked by the microstructure of cross-sections through the notch. Following notch preparation, the specimens were surface ground to proper finished dimensions.

Ten specimens per condition were tested to ±0.5 ft-lb in a Tinius-Olsen Change-o-Matic impact tester, using a correction curve derived from calibration according to ASTM Methods E 23 – 56 T. The results were statistically analyzed by calculating the deviation of the mean, $3\,\sigma_{\bar{x}}$, and the fracture appearance was examined under a binocular microscope. For low-temperature testing, the specimens were cooled for 15 min after the cessation of boiling in an insulated container, using an acetone - dry ice mixture for −100 F and liquid nitrogen for −320 F. Elevated temperature impact tests at 300 to 1100 F were conducted from an adjacent resistance furnace, adjusted to compensate for the temperature drops observed in a thermocouple-equipped specimen during trial transfers.

Standard R. R. Moore-type fatigue specimens were machined from $\frac{5}{8}$-in. round bar stock to the dimensions shown in Fig. 3(*a*), of the ASM Metals Handbook.[10] After heat treatment 0.040 in. was removed, followed by a standard longitudinal polish, and stress relief of 2 hr at 750 F. Testing was accomplished on a 200 in.-lb Baldwin-Lima-Hamilton R. R. Moore-type machine. Cycling speeds were 5000 to 10,000 cpm for the long life tests and 1000 to 5000 cpm for the short duration tests at high stress levels. Sufficient tests were run to obtain only a preliminary survey of the *S-N* curves, with no attempt at statistical analysis of the results.

---

[8] Tentative Recommended Practice for Short-Time Elevated Temperature Tension Tests of Materials (E 21 – 58 T), 1958 Book of ASTM Standards, Part 3, p. 217.

[9] Tentative Methods for Notched Bar Impact Testing of Metallic Materials (E 23 – 56 T), 1958 Book of ASTM Standards, Part 3, p. 69.

[10] ASM Metals Handbook, Am. Soc. Metals (1948).

TABLE III.—TENSILE PROPERTIES OF 5 PER CENT CHROMIUM STEELS AT VARIOUS TEMPERED HARDNESS LEVELS.

Standard 0.505 in. specimens, neutral austenitized 30 min at 1825–1850 F, air cooled, and triple tempered 2 + 2 + 2 hr at indicated temperatures. Tested in triplicate.

Steel	Tempering Temperature, deg Fahr (2 + 2 + 2 hr)	Rockwell Hardness, C Scale	Yield Strength, psi					Ultimate Tensile Strength, psi	True Fracture Stress, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Modulus of Elasticity psi
			Proportional Limit	0.01 per cent Offset	0.02 per cent Offset	0.1 per cent Offset	0.2 per cent Offset					
X2	950	51.6	106 000	126 000	142 000	184 000	206 000	262 000	395 000	12.1	50.1	31.4 × 10⁶
	1000	50.8	105 000	132 000	144 000	186 000	208 000	251 000	399 000	13.1	55.5	31.5
	1050	48.4	133 000	140 000	151 000	188 000	204 000	234 000	360 000	13.1	58.0	31.7
	1100	41.1	123 000	133 000	140 000	164 000	172 000	192 000	286 000	14.4	54.7	31.6
	1200	28.0	67 000	85 000	92 000	103 000	107 000	131 000	209 000	18.2	56.6	33.9
X3	950	55.1	120 000	136 000	148 000	200 000	228 000	295 000	392 000	9.9	33.9	30.0
	1000	53.1	128 000	143 000	158 000	207 000	229 000	276 000	386 000	10.0	42.5	30.7
	1050	50.2	131 000	146 000	160 000	200 000	220 000	255 000	353 000	10.0	43.2	31.8
	1100	44.6	122 000	137 000	145 000	170 000	181 000	208 000	297 000	12.4	48.3	31.7
	1200	29.2	58 000	75 000	85 000	101 000	106 000	136 000	212 000	17.1	54.6	34.5
VJ	950	57.0	107 000	140 000	154 000	210 000	241 000	311 000	324 000	6.6	27.0	30.5
	1000	54.0	107 000	142 000	159 000	211 000	235 000	290 000	361 000	7.8	28.1	30.3
	1050	52.0	119 000	148 000	162 000	206 000	226 000	268 000	342 000	8.5	31.9	30.9
	1100	46.0	114 000	141 000	150 000	178 000	189 000	221 000	291 000	11.2	38.5	30.9
	1200	33.0	84 000	101 000	107 000	119 000	124 000	154 000	212 000	14.1	41.2	32.1
VJ-VM	950	56.0	129 000	144 000	158 000	211 000	238 000	306 000	391 000	8.7	30.4	30.6
	1000	53.9	122 000	143 000	162 000	209 000	232 000	284 000	418 000	11.4	50.8	32.3
	1050	50.8	126 000	143 000	156 000	199 000	217 000	257 000	377 000	12.9	52.2	31.9
	1100	45.9	121 000	140 000	152 000	176 000	186 000	218 000	323 000	13.6	52.4	31.2
	1200	32.3	60 000	78 000	90 000	107 000	114 000	147 000	240 000	17.5	58.8	32.5
X5	950	58.0	142 000	175 000	197 000	250 000	274 000	329 000	373 000	5.3	13.6	30.7
	1000	55.5	142 000	167 000	186 000	236 000	261 000	305 000	361 000	6.4	22.5	31.5
	1050	52.2	135 000	154 000	173 000	219 000	237 000	274 000	329 000	7.4	25.8	31.6
	1100	47.4	125 000	141 000	152 000	181 000	193 000	231 000	289 000	9.2	30.7	32.3
	1200	35.0	61 000	85 000	98 000	117 000	124 000	158 000	219 000	13.3	40.8	31.8

EXPERIMENTAL RESULTS

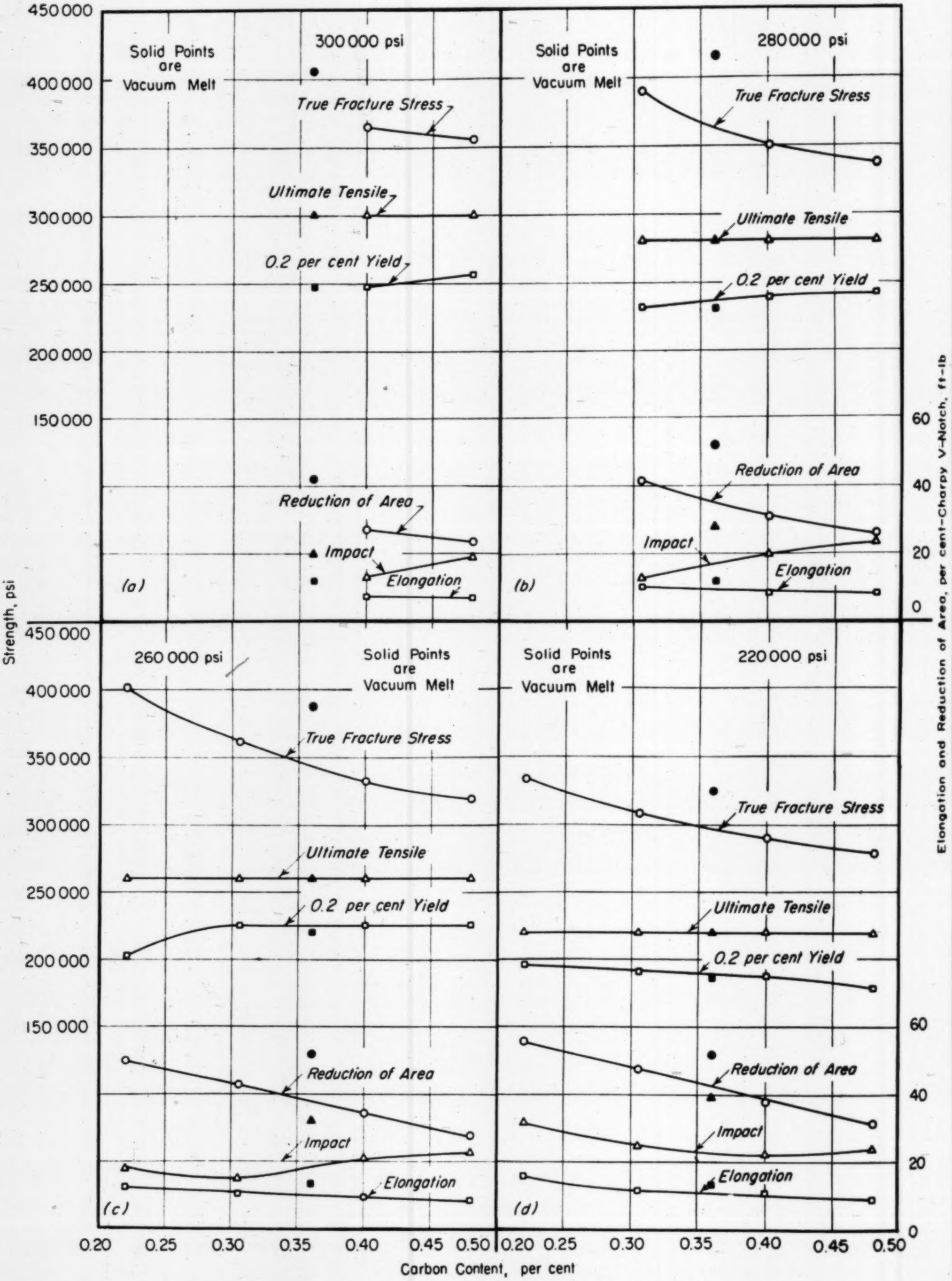
Room-Temperature Properties:

Tensile and Impact Properties.—Average tensile properties of the five steels from triplicate tests at each of five tempered hardness levels are summarized in Table III. In addition to the conventional property data, values were determined for the modulus of elasticity, true fracture stress (breaking load divided by final area), and yield strengths at proportional limit, 0.01, 0.02, 0.1, and 0.2 per cent offset. Typical autographic stress-strain curves and corresponding true stress - true strain curves have been reported (20) for various tempered hardness levels in the 0.40 per cent carbon VJ steel. Stress-strain curves for the other four steels were similar in appearance, the principal differences being the amount of strain before fracture corresponding to the elongation values. The 0.50 per cent carbon X5 steel also showed a slightly lower rate of increase in load per unit of strain, from the 0.2 per cent yield to the maximum load, indicating a slightly decreased rate of strain hardening. Calculation of the strain-hardening exponent and the rate of strain hardening at various tempered hardness levels in the 0.40 per cent carbon steel indicated a relatively constant value for

TABLE IV.—CHARPY IMPACT STRENGTH OF 5 PER CENT CHROMIUM STEELS AT VARIOUS ULTIMATE STRENGTH LEVELS.

Standard V-notch Charpy specimens, neutral austenitized 30 min at 1825 to 1850 F, air cooled, and triple tempered 2 + 2 + 2 hr at indicated temperatures. Ten tests per condition.

Steel	Tempering Temperature, deg Fahr	Rockwell Hardness, C Scale	Ultimate Tensile Strength, psi	V-Notch Charpy Impact Strength, ft-lb		
				Average	Range	$3\sigma_{\bar{x}}$
X2	950	51.7	262 000	17.5	11.0 to 21.5	2.6
	1000	50.2	243 000	19.7	15.5 to 22.0	2.2
	1050	48.3	234 000	25.9	20.5 to 32.0	2.6
	1100	40.9	192 000	41.5	37.0 to 46.0	2.1
	1200	33.0	154 000	81.4	78.0 to 90.0	3.4
X3	950	54.8	290 000	12.3	11.0 to 14.0	1.0
	1000	52.5	272 000	12.7	11.5 to 14.0	0.9
	1050	50.1	255 000	15.7	14.0 to 17.0	1.0
	1100	44.5	208 000	29.2	26.5 to 30.5	1.1
	1200	29.4	136 000	61.4	59.0 to 63.5	1.4
VJ	950	56.5	311 000	11.2	7.5 to 13.5	1.3
	1000	55.4	302 000	15.7	13.0 to 18.0	1.6
	1050	51.4	263 000	20.4	16.5 to 23.0	1.9
	1100	46.7	220 000	22.3	21.0 to 23.5	1.0
	1200	33.0	154 000	29.5	27.5 to 31.5	2.2
VJ-VM	950	55.0	294 000	19.0	15.0 to 21.0	2.2
	1000	52.9	277 000	24.9	20.0 to 34.0	4.4
	1050	50.3	246 000	33.1	30.0 to 35.5	1.8
	1100	44.5	207 000	39.8	38.0 to 41.5	1.1
	1200	30.5	142 000	92.9	89.0 to 95.5	1.8
X5	950	57.6	318 000	14.5	12.0 to 18.0	2.0
	1000	54.9	294 000	18.6	15.0 to 21.0	1.8
	1050	51.8	270 000	21.2	19.0 to 25.0	2.0
	1100	47.2	231 000	23.5	21.5 to 26.0	1.3
	1200	34.2	163 000	41.7	40.0 to 43.5	1.0



(*a*) 300,000 psi ultimate strength. (*b*) 280,000 psi ultimate strength.
(*c*) 260,000 psi ultimate strength. (*d*) 220,000 psi ultimate strength.

FIG. 4.—Effect of Carbon Content on Tensile and Impact Properties of 5 per cent Chromium Steels at Specified Ultimate Tensile Strength Levels.

the latter over a broad hardness range from Rockwell hardness C 30 to 57.

The Charpy V-notch properties of the five steels, tempered to the same hardness and strength levels as the tension specimens, are summarized in Table IV. The use of 10 tests per condition resulted in a statistical deviation of the mean, $3\sigma_{\bar{x}}$, of less than ±2 ft-lb for a majority of the conditions. Five additional specimens of each steel were also tested in the as air-cooled, untempered condition. The results were quite high: 37.3 ft-lb for X2; 24.0 ft-lb for X3; 25.5 ft-lb for VJ-VM; and 12.4 ft-lb for X5, with low scatter similar to the tempered specimens. High untempered toughness is, of course, important to crack-free welding and heat treatment.

By plotting the tensile and impact properties against the tempering temperature for each steel (Fig. 3(*a* to *e*)), it is possible to study the various combinations of properties available in each steel over its entire spectrum of strength capability. Typical for the 5 per cent chromium steels is the absence of any embrittlement phenomenon, with multiple tempering beyond the secondary hardness peak. At increasing tempering temperatures, steadily decreasing strength properties are obtained with corresponding improvements in ductility and toughness. Furthermore, as the carbon content is increased, the strength values at a given tempering temperature also increase, and the ductility properties show a trend to lower values.

The trends in true fracture stress shown by the vacuum-melted 0.40 per cent carbon VJ-VM in Fig. 3(*d*) and the 0.20 per cent carbon X2 in Fig. 3(*a*) are noteworthy. The higher ductility provided by vacuum melting at the 0.40 per cent carbon level or by reducing the carbon content to 0.20 per cent, produced the highest true fracture-stress values among the five steels. High values for this property may be of significance to certain pressure vessel applications where high burst strengths are of primary importance and yield strength assumes secondary significance.

In Fig. 4(*a* to *d*) the tensile and impact properties of the various steels have been plotted against carbon content for constant ultimate tensile levels of 300,000, 280,000, 260,000, and 220,000 psi respectively. The 300,000 psi tensile level (Fig. 4(*a*)) was obtainable only with carbon levels of 0.36 to 0.48 per cent. Within these limits, yield and impact strengths increased slightly, and ductility decreased, with increasing carbon content. The vacuum melted VJ-VM, indicated by the solid points, showed pronounced improvement in ductility. At the 280,000 psi ultimate tensile level (Figure 4(*b*)), the 0.30 per cent carbon material was added to the group having this strength capability. The consistent trend of slightly increased yield strength and improved impact strength, with reduced ductility and true fracture stress, were again noted as the carbon content increased. The superiority of the vacuum melted VJ-VM in ductility, toughness, and true fracture stress was likewise reemphasized.

All five steels achieved the strength capability of the 260,000 psi ultimate tensile level shown in Fig. 4(*c*). At this level, the yield strength is nearly constant with carbon content, except at the lowest carbon level which is slightly undertempered. The ductility values decrease almost linearly with increasing carbon content, while the impact strength shows a slight reversal in its former trend of decreasing with lower carbon content and shows a slight upturn at the 0.20 per cent carbon level. It is noteworthy that the vacuum melted steel VJ-VM again exhibits marked superiority, the ductility values and true fracture stress being about equivalent to a reduction in carbon content of 0.14 per cent, while the impact strength lay considerably above all values obtained. At the still lower 220,000 psi ultimate tensile level (Fig. 4(*d*)), the 0.20 per cent carbon X2 steel came fully into its realm, and all properties showed a consistent decrease with increasing carbon content. Again, the vacuum melted material gave values considerably above those expected for its carbon content.

TABLE V.—SHEET TENSILE PROPERTIES OF 5 PER CENT CHROMIUM STEELS AT VARIOUS TEMPERED HARDNESS LEVELS.

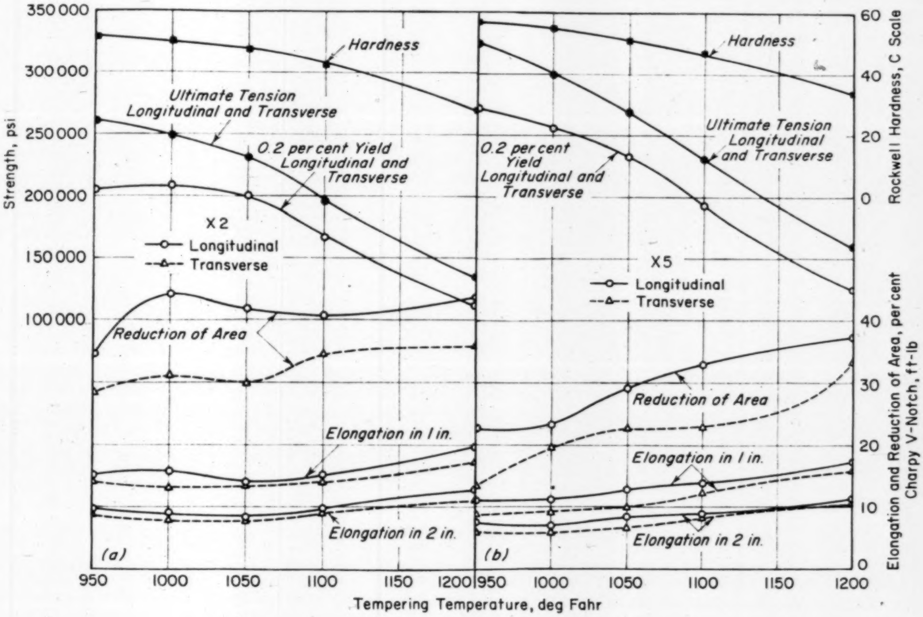
Standard 0.100 by 0.5-in. sheet specimens, neutral austenitized 30 min at 1825 to 1850 F, air cooled and triple tempered 2 + 2 + 2 hr at indicated temperatures. Triplicate tests both longitudinal and transverse to long dimension of sheet.

Steel	Tempering Temperature, deg Fahr	Rockwell Hardness, C Scale	Yield Strength 0.2 per cent Offset, psi	Ultimate Tensile Strength, psi	Elongation in 2 in., per cent		Elongation in 1 in., per cent		Reduction of Area, per cent	
					Longitudinal	Transverse	Longitudinal	Transverse	Longitudinal	Transverse
X2	950	51.3	204 000	261 000	9.7	8.6	15.2	14.2	34.5	28.5
	1000	49.9	208 000	248 000	9.3	7.8	15.7	13.2	44.1	31.1
	1050	47.5	200 000	231 000	8.3	7.6	14.0	13.3	41.7	29.8
	1100	42.4	174 000	196 000	9.6	8.9	15.2	14.0	40.7	34.5
	1200	27.8	112 000	134 000	12.8	11.1	19.7	17.2	43.6	35.9
X3	950	53.7	230 000	283 000	6.7	6.6	11.3	11.0	27.4	23.6
	1000	52.1	226 000	265 000	6.9	6.3	11.8	10.3	32.5	24.3
	1050	49.3	209 000	242 000	6.8	5.8	13.0	11.0	35.0	26.1
	1100	44.7	182 000	211 000	7.2	6.8	11.7	11.8	36.8	31.1
	1200	28.7	108 000	137 000	12.5	10.9	19.5	17.0	41.2	35.1
VJ	950	55.9	240 000	309 000	6.7	5.5	11.2	8.8	26.1	19.0
	1000	54.1	233 000	287 000	6.0	5.1	9.7	8.2	37.9	28.2
	1050	51.3	220 000	263 000	7.1	6.4	11.5	10.3	31.7	26.1
	1100	47.4	196 000	230 000	8.2	7.2	13.2	11.6	33.0	27.9
	1200	32.4	123 000	154 000	11.1	9.9	17.2	15.6	38.7	33.1
X5	950	57.2	273 000	326 000	7.4	5.7	11.0	8.5	22.5	13.2
	1000	55.0	256 000	299 000	6.9	5.7	11.2	9.0	23.2	19.6
	1050	51.1	232 000	268 000	8.4	6.5	12.8	9.8	29.1	22.6
	1100	47.1	192 000	231 000	8.8	8.1	13.8	12.2	32.8	22.7
	1200	33.4	124 000	159 000	11.3	10.6	17.3	15.8	37.4	33.1

Sheet Properties.—In the form of 0.125-in. thick sheet, the four carbon levels gave the sheet tensile properties shown in Table V from triplicate tests in each direction. The 0.2 per cent yield strength and ultimate strength values from the six specimens representing both directions were equivalent within the limits of experimental error and are each reported as single values. Although steels of this type are traditionally cross rolled, slight differences still appear in the longitudinal and transverse ductility properties due to the final reduction to finish size, and are therefore reported separately. Comparison of these data with the bar tensile properties of Table III indicates values of similar magnitude, for both material forms after a given hardening and tempering treatment.

A few words might be inserted here regarding sheet tension testing, from experience with several thousand tests run in these and other laboratories. Considerable confusion has arisen in the evaluation of sheet materials for pressure vessel applications due to inconsistencies in the measurement of sheet yield strengths. Extensometers clamped to the gage length of the specimen, which are of the nonaveraging type for sheet, tend to give low readings (as much as 30,000 psi below the true value) due to warpage of the specimens during preparation. If the specimens are straightened before testing, residual stresses on one surface are added to the testing stresses, and the material yields on this surface at a relatively low value. If the specimen is placed in the grips in a warped condition, similar high stresses are produced on one surface. When, on the other hand, a deflectometer or similar instrument is used to measure crosshead movement, measurement errors tend to give yield strength values slightly higher than true. From careful sheet tensile studies, it has been found that sheet tensile yield strengths of properly heat-treated specimens are practically identical with bar tensile yield strengths from the same steel.

Elongation measurement in sheet tension testing is also quite misleading and should be a consideration for future standards revisions. It has been demonstrated by theoretical and experimental work on metallic materials for many years that, for equivalent elongation values, the gage length of the specimen should be approximately equal to 4.5 times the square root of the area. Thus, in bar tension, creep, and rupture testing, specimens 0.505 in. in diameter employ a 2-in. gage length, 0.252-in. specimens a 1-in. gage length, and so on. The standard ASTM sheet tension specimen, however, employs a 2-in. gage length, with an area that is generally equivalent to or smaller than the 0.252-in. round bar. The resulting per cent elongation in 2 in. values for sheet are unnecessarily low and mislead designers into thinking that a material in sheet form is less ductile than the corresponding material in bar or forging. To offset this problem, our laboratory has established a practice of marking off an



(*a*) 0.20 per cent carbon (*b*) 0.50 per cent carbon

FIG. 5.—Representative Sheet Tensile Properties of 5 per cent Chromium Steels. 0.125-in thick sheet, tested both parallel and transversely to its long dimension.

additional 1-in. gage length in the center of the sheet tension specimen and reporting elongations in 1 in., as well as 2 in. A plot of these data indicate that per cent elongation in 1 in. is approximately 1.6 times the per cent elongation in 2 in.

Values for reduction of area have also been included in Table V, although subject to some error due to the 45-deg shear valleys that appear on the surfaces of the sheet during neckdown and vary in configuration with the material and the tempered hardness level. The approximate values appear to be quite comparable to those of corresponding bar stock.

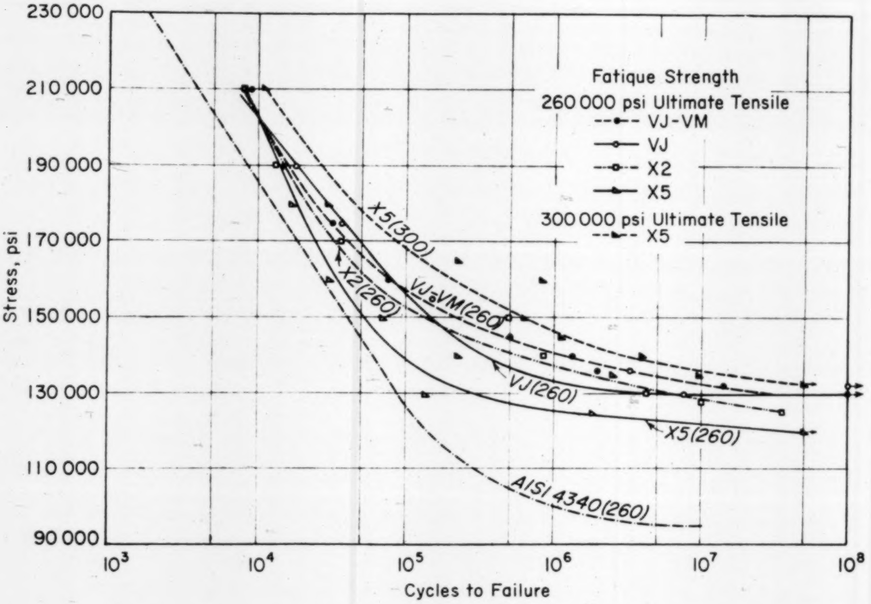


FIG. 6.—Preliminary *S-N* Fatigue Curves from R.R. Moore Rotating-Beam Tests on 5 per cent Chromium Steels.

Average curve for AISI 4340 steel, heat treated to the same strength level and identically tested, is included for comparison (**21**).

The sheet tensile properties of the two new materials, 0.20 per cent carbon X2 and 0.50 per cent carbon X5, are shown graphically in Figs. 5(*a*) and (*b*). The sheet properties of the 0.40 per cent carbon VJ, in both the unwelded and welded conditions, have previously been reported (**17,20**).

The 0.20 per cent carbon X2 steel exhibits excellent ductility at ultimate strength levels around 250,000 psi and yield strengths above 200,000 psi. With a higher strength potential than AISI 4130 steel at about 0.10 per cent less carbon content, this material may offer promise for applications requiring maximum ductility and weldability with a minimum of preheat and postheat operations. The weld depositing characteristics of the 0.40 per cent carbon VJ have been quite universally reported as excellent, and similar performance might be expected from a lower carbon version of the same material.

The 0.50 per cent carbon X5 steel offers higher ultimate strength and yield strength potentials than available in the 0.40 per cent carbon range presently used, and may offer promise for ultra-high-strength strip, pressure vessels being drawn or spun to avoid longitudinal seams, formed parts, and precision welded parts of highest strength capability.

Fatigue Properties.—Preliminary fatigue curves, using best average lines, are plotted for four of the steels heat treated to the 260,000 psi tensile level and for X5 at the 300,000 psi ultimate tensile level (Fig. 6). For comparison, the average curve for AISI 4340, also heat treated to 260,000 psi tensile strength and identically tested, as reported by Cummings, Stulen, and Schulte (21), has been included. The 130,000-psi endurance limit for the 0.40 per cent carbon VJ at the 260,000 psi tensile level has been confirmed by tension-tension fatigue tests on specimens removed from 6-in. square billet stock (22).

The curves for the X2, VJ, and VJ-VM fell within essentially the same band. However, the X2 failed to show as high or as definite an endurance limit as the other two steels. The 0.50 per cent carbon X5, when tempered to a lower strength level than that for which it would be most useful, indicated lower fatigue characteristics than the other 5 per cent chromium steels. However, when heat treated to the 300,000 psi ultimate tensile level, it exhibited somewhat higher fatigue properties than any of the compositions tested.

The 35 per cent difference in fatigue endurance limit between the 0.40 per cent carbon 5 per cent chromium steel and the 0.40 per cent carbon AISI 4340 steel when both are heat treated to the 260,000 psi ultimate tensile level, may be related to the finding of Frankel, Bennett, and Pennington (9), that retained austenite lowers the fatigue properties of high-strength steel (less than 1 per cent in the 5 per cent chromium steel). Other factors that may contribute to this difference are more complete stress relief during tempering (1050 F for the 5 per cent chromium steel *versus* 450 F for the 4340 steel), and differences in melting practice. The close relationship of the vacuum-melted VJ-VM and the air-melted VJ curves would indicate that the air-melted quality of the latter steel was sufficient to minimize the effect of inclusions on its fatigue properties.

Notch Transition Properties:

For a preliminary comparison of notch properties, impact transition curves were obtained from elevated temperatures through room temperature to −320 F, with all specimens tempered to the same 260,000 psi ultimate strength level. Ten tests per condition again gave statistical deviations of less than ±2 ft-lb for most values, as shown in Table VI. Relative percentages of shear and cleavage fracture were estimated under the binocular microscope. These ratings were necessarily quite approximate because the extremely fine grain size prohibited sharp differentiation between the modes of fracture.

In Fig. 7, these data have been plotted for the temperature range −320 F to +500 F of interest to transition studies. As indicated in previously reported investigations, the higher carbon VJ, VJ-VM, and X5 steels show no sharply defined transition temperatures, maintaining values of 15 ft-lb at −100 F and appearing to plot smoothly to −320 F. The fracture appearance also indicated no sharp transition and retained appreciable percentages of fibrous fracture to −320 F.

The changes in impact strength between 300 F and 70 F for the low carbon X2 and X3 steels are suggestive of a transition in this region. However, the absolute values of impact strength were still relatively high at the subzero temperatures, and their fracture appearance

TABLE VI.—EFFECT OF TEST TEMPERATURE ON CHARPY IMPACT STRENGTH OF 5 PER CENT CHROMIUM STEELS HEAT TREATED TO 260,000 PSI ULTIMATE TENSILE STRENGTH LEVEL.

Standard V-notch Charpy specimens, neutral austenitized 30 min at 1825 to 1850 F, air cooled, and triple tempered 2 +2 + 2 hr at indicated temperatures. Ten tests per condition.

Test Temperature, deg Fahr	V-Notch Charpy Impact Strength, ft-lb														
	Steel X2 Tempered at 950 F			Steel X3 Tempered at 1030 F			Steel VJ Tempered at 1055 F			Steel VJ-VM Tempered at 1030 F			Steel X5 Tempered at 1060 F		
	Average	Range	$3\sigma_{\bar{x}}$	Average	Range	$3\sigma_{\bar{x}}$	Average	Range	$3\sigma_{\bar{x}}$	Average	Range	$3\sigma_{\bar{x}}$	Average	Range	$3\sigma_{\bar{x}}$
−320	5.1	4.5 to 6.0	0.3	5.3	4.0 to 8.0	1.0	5.7	5.0 to 6.5	0.4	6.8	5.0 to 8.0	0.9	7.9	6.0 to 10.5	1.4
−100	10.4	6.0 to 15.5	3.5	11.0	6.0 to 16.0	3.8	15.3	13.0 to 19.0	1.7	15.7	14.0 to 17.0	0.9	16.7	15.0 to 17.5	0.7
70	17.5	11.0 to 21.5	2.6	13.1	9.0 to 18.5	2.5	22.0	18.0 to 25.5	2.2	25.3	21.5 to 28.0	1.9	21.2	19.0 to 23.0	1.0
300	40.5	39.0 to 44.0	...	31.2	29.0 to 33.0	1.6	27.1	24.0 to 30.0	2.0	39.4	37.0 to 41.5	1.4	24.6	21.5 to 27.0	1.6
500	41.1	39.0 to 44.0	...	32.5	30.0 to 35.0	1.4	30.4	28.5 to 32.0	1.2	43.9	41.0 to 47.0	1.8	25.1	24.0 to 26.5	0.9
700	39.3	37.5 to 42.0	1.5	34.0	33.0 to 35.0	0.8	32.1	30.0 to 34.0	1.1	47.4	46.0 to 49.0	0.9	26.0	24.5 to 27.0	0.9
900	39.2	37.0 to 40.0	0.9	31.6	30.0 to 35.0	1.3	29.3	28.0 to 31.0	1.1	48.2	47.0 to 49.0	0.7	26.2	25.0 to 28.0	0.8
1000	35.8	34.0 to 37.0	1.2	29.5	27.0 to 30.5	0.9	30.5	29.0 to 32.5	1.3	48.5	47.0 to 50.0	1.0	25.7	24.0 to 28.0	1.1
1100	35.4	35.0 to 36.5	0.4	29.1	28.0 to 31.5	0.9	33.2	30.5 to 34.5	1.0	48.5	47.0 to 50.5	1.2	27.1	25.5 to 28.0	0.7

ratings at −320 F were 45 to 55 per cent fibrous.

Thus, unlike the bar and sheet tension tests and the room temperature impact tests, we find here a noticeable change on going from 0.40 to 0.30 per cent carbon, in which the lower carbon materials exhibit some tendency for impact transition behavior similar to low alloy steels. Relatively good crack propagation characteristics of the 0.40 per cent carbon VJ steel have been reported from several laboratories (**23–25,29–32**), and high notch tensile properties have also been reported for this material (**5,26**). However, in instances where a lack of neutral hardening conditions has resulted in some surface decarburization, the crack propagation characteristics have been less favorable. This difference in results may be related to the above noted change in impact transition properties with carbon content.

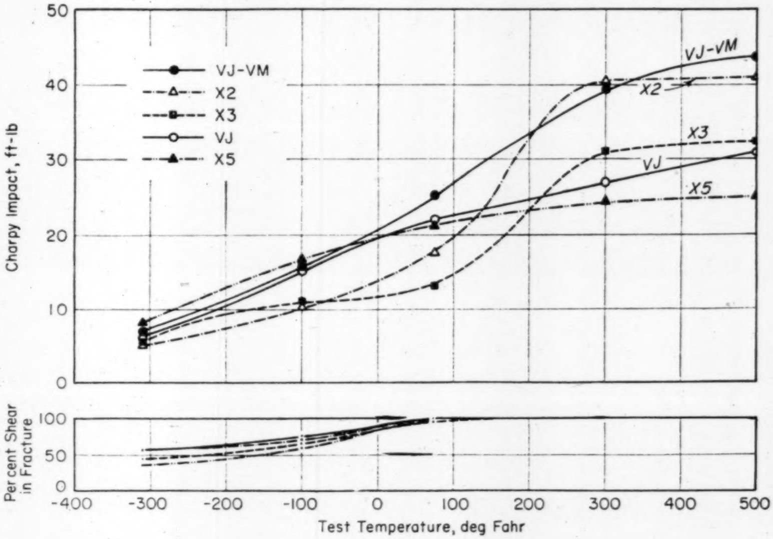


FIG. 7.—Charpy V-Notch Impact Transition Curves, Using Both Energy and Fracture Appearance Criteria, for 5 per cent Chromium Steels.

Specimens heat treated to the 260,000 psi ultimate tensile strength level and tested between −320 and +500 F.

Elevated Temperature Tensile and Impact Properties:

The room temperature strength level selected for a given elevated temperature application should be based on the operating temperature and operating time during which stability is desired. For example, steel VJ tempered at 1050 F to 260,000 psi ultimate tensile strength should be structurally stable for 10 sec at 1330 F, 1 hr at 1100 F, 100 hr at 975 F, 1000 hr at 900 F, or 1,000,000 hr (125 yr) at 750 F. These times, which can be readily calculated from the parameter shown in Fig. 1, are based on the time-temperature relationships in the tempering of martensite devised by Hollomon and Jaffe (**19**) and studied for numerous medium to high alloy steels by Roberts, Grobe, and Moersch (**27**). They indicate maximum heating times for retention of room temperature properties on again cooling to room temperature, providing surface changes, creep, or other phe-

TABLE VII.—ELEVATED TEMPERATURE TENSILE PROPERTIES OF 5 PER CENT CHROMIUM STEELS HEAT TREATED TO THE 260,000 PSI ULTIMATE TENSILE STRENGTH LEVEL.

Standard 0.505 in. specimens, neutral austenitized 30 min at 1825 to 1850 F, air cooled, and triple tempered 2 + 2 + 2 hr at indicated temperatures. Two to four tests per condition, per ASTM Recommended Practice E 21 – 58 T.

Steel	Tempering Temperature, deg Fahr (2 + 2 + 2 hr)	Test Temperature, deg Fahr	Yield Strength, psi					Ultimate Tensile Strength, psi	True Fracture Stress, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Modulus of Elasticity, psi
			Proportional Limit	0.01 per cent Offset	0.02 per cent Offset	0.1 per cent Offset	0.2 per cent Offset					
X2	950	room temperature	106 000	126 000	142 000	184 000	206 000	262 000	395 000	12.1	50.1	31.4 × 10⁶
		800	84 000	111 000	124 000	160 000	181 000	230 000	339 000	11.5	49.9	25.8
		900	84 000	99 000	109 000	146 000	167 000	220 000	292 000	12.1	43.3	26.4
		1000	69 000	86 000	100 000	139 000	158 000	203 000	261 000	10.3	45.4	25.3
		1100	57 000	66 000	74 000	106 000	124 000	156 000	168 000	11.0	44.2	22.8
X3	1030	room temperature	158 000	178 000	185 000	211 000	226 000	259 000	362 000	11.0	46.8	30.1
		800	73 000	104 000	117 000	145 000	173 000	217 000	314 000	11.6	50.5	27.6
		900	69 000	79 000	95 000	142 000	162 000	201 000	294 000	13.5	54.7	26.1
		1000	67 000	83 000	96 000	132 000	149 000	184 000	252 000	13.2	57.1	25.1
		1100	53 000	67 000	76 000	108 000	125 000	154 000	172 000	11.5	39.2	21.0
VJ	1050	room temperature	146 000	165 000	174 000	212 000	228 000	263 000	335 000	8.3	34.6	31.1
		800	70 000	96 000	115 000	156 000	175 000	215 000	309 000	13.3	50.8	26.3
		900	92 000	106 000	116 000	149 000	164 000	197 000	280 000	13.7	53.6	24.6
		1000	63 000	83 000	93 000	128 000	143 000	176 000	237 000	14.0	54.5	23.5
		1100	43 000	53 000	60 000	88 000	107 000	138 000	162 000	14.4	56.3	21.6
VJ-VM	1030	room temperature	132 000	155 000	167 000	205 000	224 000	265 000	391 000	11.9	53.2	31.1
		800	67 000	87 000	105 000	152 000	173 000	219 000	369 000	14.8	63.6	28.1
		900	70 000	87 000	101 000	145 000	165 000	205 000	329 000	15.9	64.7	26.8
		1000	66 000	79 000	90 000	129 000	147 000	186 000	267 000	15.3	62.0	24.5
		1100	48 000	63 000	73 000	104 000	121 000	151 000	181 000	13.9	57.0	22.0
X5	1060	room temperature	130 000	143 000	155 000	193 000	212 000	260 000	335 000	8.3	26.9	31.6
		800	97 000	112 000	125 000	161 000	181 000	220 000	299 000	10.8	43.2	28.6
		900	70 000	96 000	105 000	142 000	160 000	199 000	275 000	12.8	49.0	27.2
		1000	59 000	69 000	81 000	118 000	137 000	176 000	236 000	15.4	56.0	24.3
		1100	41 000	52 000	60 000	90 000	106 000	140 000	173 000	16.7	58.0	20.7

nomena have not otherwise altered the material.

For a comparison of elevated temperature properties, all five steels were heat treated to the same 260,000 psi ultimate tensile level, which was the highest strength achievable by the lowest carbon material. Due to the similarity in their tempering curves, these 5 per cent chromium steels could be heat treated to the same room temperature strength by adjusting their tempering temperatures over a narrow 110 F range. The tempering temperature used to achieve the 260,000 psi tensile level and the corresponding elevated temperature properties at 800, 900, 1000, and 1100 F are summarized in Table VII. As in the tension tests at room temperature, two to four specimens were run per condition, elevated temperature stress-strain curves were recorded, and the various engineering yield strengths reported in addition to true fracture stress and elastic modulus. Elevated temperature stress-strain curves through 0.2 per cent yield strength, and secant and tangent modulus data have already been reported for the 0.40 per cent carbon VJ (**20,28**). Elevated temperature impact specimens in this temperature range, in addition to those used in the aforementioned transition studies, were similarly heat treated to the 260,000 psi ultimate tensile level, and were included in Table VI.

To study elevated temperature tensile properties at maximum obtainable strength levels, additional sets of specimens were prepared from the 0.40 per cent carbon VJ and the 0.50 per cent carbon X5, heat treated to the 311,000 and 329,000 psi ultimate tensile levels respectively. As shown in Table VIII, these steels gave ultimate tensile values for 30 min at 1000 F of 216,000 and 229,000 psi respectively. With accelerated heating and testing rates, they would be expected to retain these strengths to considerably higher temperatures. These high 1000 F strengths were accompanied by favorable ductility values of 9 to 12 per cent elongation and 34 to 42 per cent reduction of area.

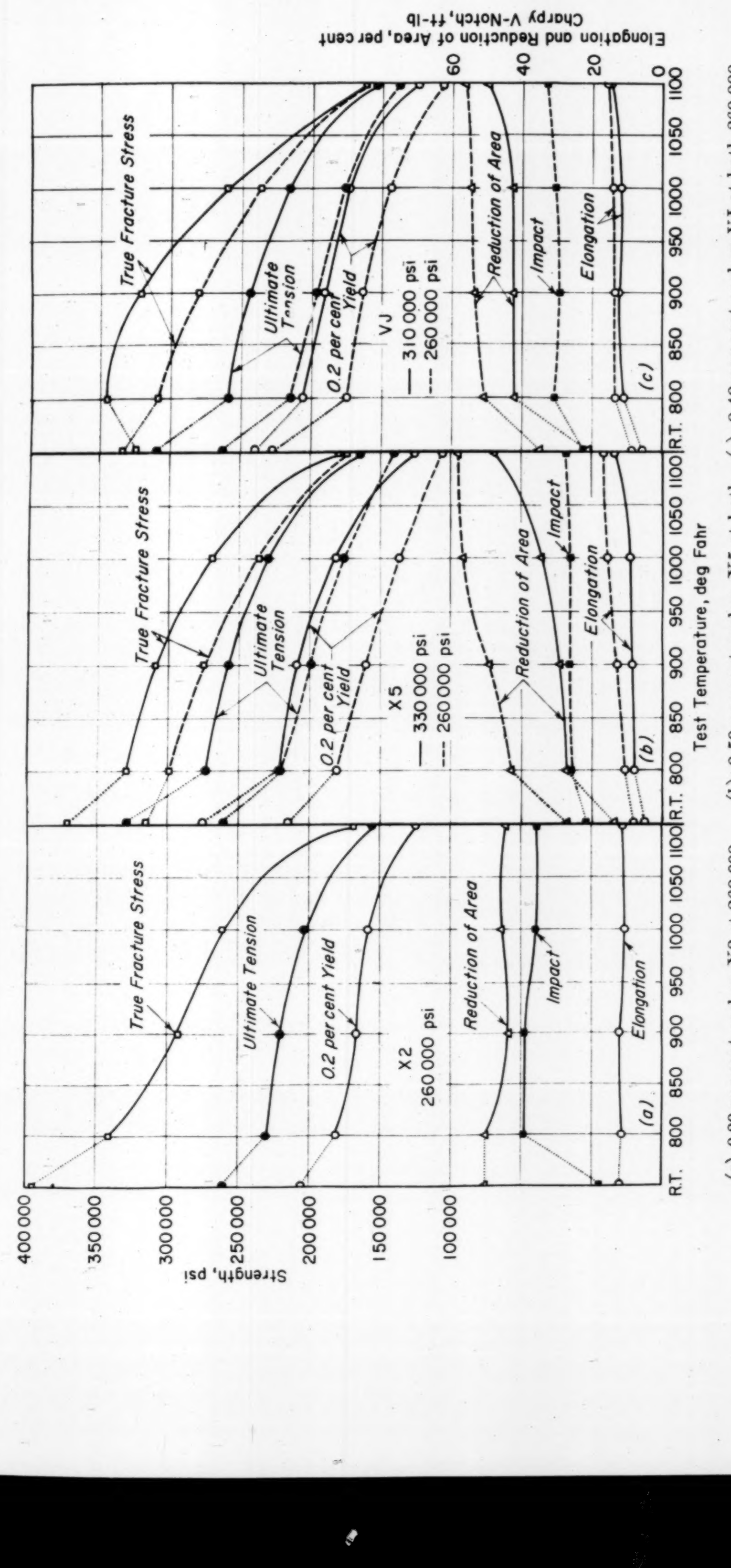
Figures 8(*a*, *b*, and *c*) show typical variations in tensile and impact properties with test temperature for X2 at the 260,000 psi room temperature ultimate tensile level, and VJ and X5 at both the 260,000 psi ultimate and the 310,000-plus psi ultimate tensile levels. The strength properties decrease steadily with increasing temperature, with a more rapid reduction appearing as the temperature is raised to 1100 F. The ductility and toughness properties exhibit excellent consistency across the entire test temperature range, regardless of the initial heat-treated strength. Previously published data for VJ (**17,20**) indicate that this consistency persists to lower temperatures, with no abrupt dips of the type sometimes encountered in high-strength materials.

The effect of carbon on tensile and impact properties at 800, 900, 1000, and 1100 F respectively is shown in greater detail in Figs. 9(*a*) to (*d*). In general, the values are relatively independent of carbon content when the various steels are heat treated to the same ultimate tensile strength level. However, there is a slight but consistent trend toward higher elevated temperature strength with lower carbon content, particularly at the higher test temperatures. This effect, similar to the indications of the Master Tempering Curves, probably also relates to the more highly alloyed carbides from lower carbon - higher alloy ratios as described by Kuo (**2**). From a practical viewpoint, the major elevated temperature property difference between the steels lies in the ability of the higher carbon steels to be heat treated to higher room temperature strength levels which in turn give considerably greater elevated temperature strength capabilities (Table

TABLE VIII.—ELEVATED TEMPERATURE TENSILE PROPERTIES OF VJ AND X5 HEAT TREATED TO THE 311,000 AND 329,000 psi ULTIMATE TENSILE STRENGTH LEVELS.

Standard 0.505-in. specimens, neutral austenitized 30 min at 1825 to 1850 F, air cooled, and triple tempered 2 + 2 + 2 hr at indicated temperatures. Two to four tests per condition, per ASTM Recommended Practice E 21 – 58 T.

Steel	Tempering Temperature, deg Fahr (2 + 2 + 2 hr)	Test Temperature, deg Fahr	Yield Strength, psi					Ultimate Tensile Strength, psi	True Fracture Stress, psi	Elongation in 2 in., per cent	Reduction of Area, per cent	Modulus of Elasticity, psi
			Proportional Limit	0.01 per cent Offset	0.02 per cent Offset	0.1 per cent Offset	0.2 per cent Offset					
VJ........	975	room temperature	107 000	140 000	159 000	210 000	241 000	311 000	379 000	6.6	27.0	30.5 × 10⁶
		800	79 000	122 000	136 000	183 000	207 000	259 000	346 000	10.8	42.1	27.6
		900	100 000	116 000	129 000	171 000	192 000	244 000	322 000	12.5	42.4	25.3
		1000	94 000	108 000	118 000	153 000	173 000	216 000	260 000	11.8	42.5	23.3
		1100	58 000	68 000	75 000	106 000	123 000	153 000	159 000	15.5	49.8	20.0
X5........	950	room temperature	142 000	175 000	197 000	250 000	274 000	329 000	373 000	5.3	13.6	30.7
		800	127 000	141 000	153 000	197 000	221 000	273 000	329 000	8.0	27.3	26.7
		900	111 000	131 000	144 000	185 000	209 000	257 000	309 000	8.4	29.0	24.1
		1000	89 000	110 000	122 000	161 000	181 000	229 000	268 000	9.0	34.1	24.3
		1100	55 000	67 000	73 000	108 000	126 000	164 000	178 000	13.4	47.7	22.0



(a) 0.20 per cent carbon X2 at 260,000 psi ultimate strength.

(b) 0.50 per cent carbon X5 at both 260,000 and 330,000 psi ultimate strength.

(c) 0.40 per cent carbon VJ at both 260,000 and 310,000 psi ultimate strength.

FIG. 8.—Elevated Temperature Tensile and Impact Properties of 5 per cent Chromium Steels Initially Heat Treated to Indicated Ultimate Strength Levels.

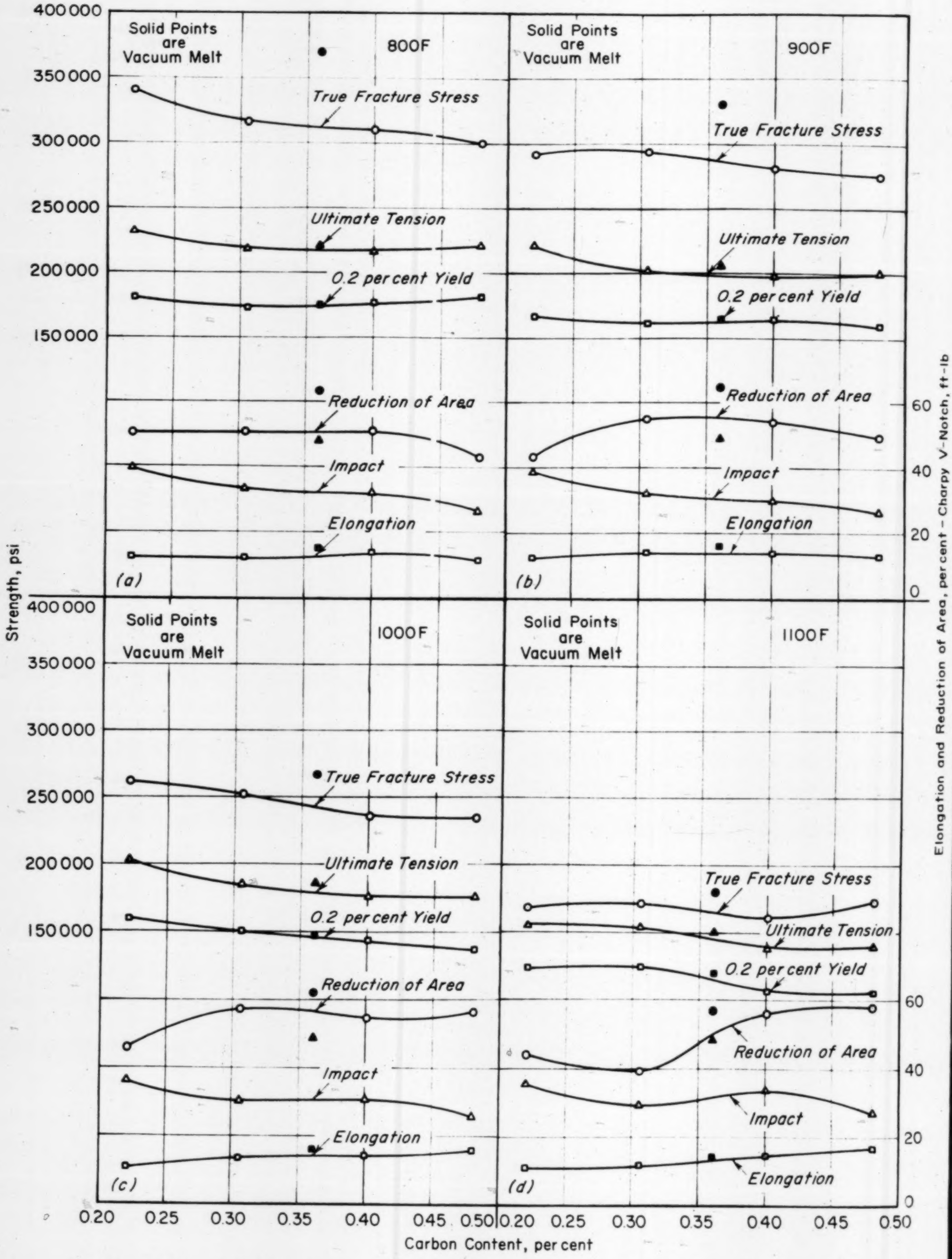


FIG. 9.—Effect of Carbon Content on Tensile and Impact Properties of 5 per cent Chromium Steels at Specified Test Temperatures.

All steels initially heat treated to the 260,000 psi ultimate tensile strength level.

VIII). Furthermore, the higher carbon steels achieve a given strength level with higher tempering temperatures which, in turn, permit higher stable operating temperatures. As with the room temperature properties, the vacuum-melted VJ-VM showed a marked superiority in ductility and toughness over all carbon levels in the air-melted steels at all test temperatures.

CONCLUSIONS

Mechanical property tests from −320 F to +1100 F indicated the following effects of carbon content and vacuum melting on the ultra-high-strength steel system containing 0.20 to 0.50 per cent carbon, 5.0 per cent chromium, 1.3 per cent molybdenum, and 0.5 per cent vanadium:

1. Vacuum melting at the 0.40 per cent carbon level produced 25 to 75 per cent improvement in ductility and impact strength at all test temperatures, overshadowing the effects of both carbon content and heat treated strength level in this respect.

2. Increasing carbon from 0.20 to 0.50 per cent raised the ultimate tensile strength capability from 260,000 to 330,000 psi, accompanied by 60 to 75 per cent decrease in ductility and little change in impact strength. As test temperature was increased to 1000 F, these strength and ductility variations with composition became less pronounced.

3. Charpy V-notch transition studies revealed no detectable transition temperature in the highest strength 0.36 to 0.50 per cent carbon steels, with appreciable percentages of shear fracture retained to −320 F. The 0.20 to 0.30 per cent carbon steels, on the other hand, indicated a moderate transition in the vicinity of room temperature. The latter could produce scatter in notch tests of the higher carbon grades if specimens are inadvertently decarburized in heat treatment.

4. Fatigue endurance limits were 120,000 to 130,000 psi for all compositions, surpassing by 25 to 35 per cent a low alloy steel similarly tested.

5. All steels exhibited similar heat treating characteristics: soft annealed properties, deep air hardenability with relatively high M_s temperatures, good impact strength in the as-hardened condition, maximum strength achieved by multiple tempering beyond a secondary hardness peak of 950 F, and a uniform tempered martensite structure free of excess carbide, ferrite, or retained austenite.

6. The lowest carbon steel, although having the highest M_s temperature with minimum retained austenite, produced the greatest response to secondary hardening. This behavior tends to confirm previous indications that secondary hardening in this system is primarily due to alloy carbide precipitation rather than to retained austenite transformation. The greater response from lower carbon is believed due to the more highly alloyed carbides from the higher alloy-to-carbon ratio in the composition.

7. The following steels from this investigation appeared promising:

(*a*) 0.20 per cent carbon X2 steel for maximum ductility with 260,000 psi ultimate and 200,000 psi yield strength capability. These strength levels surpass AISI 4130 steel, with the added advantage of 0.10 per cent lower carbon content for potentially improved formability and weldability.

(*b*) 0.50 per cent carbon X5 steel for 330,000 psi ultimate and 275,000 psi yield strength capability, with high fatigue and impact properties and moderate ductility.

(*c*) The 0.40 per cent carbon VascoJet 1000 steel, which has been used in 260,000 to 300,000 psi applications for several years, continued to exhibit the most versatile combination of properties.

8. With the present trend to higher strength levels and increasing alloy additions to achieve them, secondary hardening steels similar to those investigated offer significant advantages in fatigue strength, stress relief, hydrogen removal, and freedom from potentially unstable retained austenite.

Acknowledgment:

The authors wish to acknowledge the excellent assistance of G. E. Felbaum and R. D. Cobrando throughout the investigation, and the contributions of J. R. Schettig and C. R. Myers. They also wish to express their appreciation to G. A. Roberts and J. P. Gill for their continued guidance and support of the Vanadium-Alloys research program.

REFERENCES

(1) W. Crafts and J. Lamont, "Secondary Hardening of Tempered Martensitic Alloy Steel," *Transactions*, Am. Mining and Metallurgical Engrs., Vol. 180, p. 471 (1949).

(2) K. Kuo, "Carbide Precipitation, Secondary Hardening and Red Hardness of High Speed Steel," *Journal of the Iron and Steel Inst.*, (London), Vol. 174, p. 223 (1953).

(3) K. Kuo, "Alloy Carbides Precipitated During the Fourth Stage of Tempering," *Journal of the Iron and Steel Inst.*, (London), Vol. 184, Part 3, Nov. 1956, pp. 258–68.

(4) M. Cohen, "Retained Austenite," *Transactions*, Am. Soc. for Metals, Vol. 41, pp. 35–94 (1949).

(5) P. E. Ruff, "Hot Work Toolsteel for Aircraft," *Metal Progress*, March 1959, pp. 103–107.

(6) P. E. Ruff, "Fabricating and Finishing Toolsteels for Aircraft Parts," *Metal Progress*, April 1959, pp. 97–102.

(7) B. L. Averbach, S. G. Lorris, and M. Cohen, "Stress-Induced Transformation of Retained Austenite in Hardened Steel," *Transactions*, Am. Soc. for Metals, Vol. 44, pp. 746–56 (1952).

(8) J. Vajda, J. J. Hauser, and C. Wells, "Effect of Silicon on Transverse Properties and on Retained Austenite Content of High Strength Steels," *Transactions*, Am. Soc. for Metals, Vol. 49, pp. 517–545 (1957).

(9) H. E. Frankel, J. A. Bennett, and W. A. Pennington, "Fatigue Properties of High Strength Steels," *Transactions*, Am. Soc. for Metals, Vol. 52, Preprint No. 121 (1959).

(10) J. C. Hamaker, Jr., "Die Steel Useful for Ultra High Strength Structural Requirements," *Metal Progress*, Dec. 1956, p. 93; *Metal Progress*, Data Sheet, Dec. 1956, p. 96-B.

(11) "Vessels from Hot Die Steels," *Metal Progress*, April 1959, pp. 96-I to 96-J.

(12) "This Bolt Takes the Heat," *Steel*, May 13, 1957.

(13) "New Fastener Stays Strong at High Temperatures," *The Iron Age*, May 16, 1957, p. 110.

(14) G. A. Roberts, "Tool and Die Steels," Western Metal Congress Symposium on Aircraft Alloys for Thermal Flight up to 1200 F, *Metal Progress*, June 1957, p. 106.

(15) P. M. Unterweiser, "Tool Steel Bows to Deep Drawing," *Iron Age*, Sept. 18, pp. 83–85 (1958).

(16) L. Anselmini, "Martensitic Type High-Strength High-Temperature Steels," Report on High-Strength High-Temperature Materials for Standard Parts Symposium in Dallas, June 4–5, 1957, Aircraft Industries Assn., Washington, D. C., p. 220.

(17) J. C. Hamaker, Jr., "Martensitic 5 per cent Chromium Steel: Properties, Fabrication, and Surface Protection," Symposium on High Strength – High Temperature Materials for Standard Parts, Dallas, June 1957; Aircraft Industries Assn., Washington, D. C., p. 237.

(18) D. P. Koistinen and R. E. Marburger, *Acta Metallurgica*, Vol. 7, No. 1, Jan. 1959, pp. 59–60.

(19) J. H. Holloman and L. D. Jaffe, "Time-Temperature Relations in Tempering Steel," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 162, p. 223 (1945).

(20) VascoJet 1000 Brochure, Vanadium-Alloys Steel Co., Third Edition (1958).

(21) H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Relation of Inclusions to the Fatigue Properties of SAE 4340 Steel," *Transactions*, Am. Soc. for Metals, Vol. 49, pp. 482–510 (1957).

(22) N. Bader and E. A. Simkovich, "Tension-Tension Fatigue Properties of VascoJet 1000 5 Cr-Mo-V Steel Bar Heat Treated to 260 ksi Ultimate Tensile Strength," Republic Aviation ERMR 3875A, Jan. 20, 1958.

(23) W. F. Brown, Jr. and G. B. Espey, "Progress Report to MAB on NACA Investigations of Ultra-High Strength Sheet Alloys in the Presence of Stress Concentrations," Lewis Laboratory, Nat. Advisory Committee for Aeronautics, Cleveland, Ohio, Sept. 16, 1958.

(24) G. B. Espey, M. H. Jones, and W. E. Brown, Jr., "Th eSharp Edge Notch Tensile Strength of Several High-Strength Steel Sheet Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 837 (1959).

(25) "Crack Propagation Tests on High-Strength Steels," Armco Research Laboratories, Middletown, Ohio, Sept. 21, 1958.

(26) J. Greenspan, "Notch Tensile Properties of VascoJet 1000," AiResearch Manufacturing Co., April 10, 1958.

(27) G. A. Roberts, A. H. Grobe, and C. F. Moersch, Jr., "The Tempering of High Alloy Tool Steels," *Transactions*, Am. Soc. for Metals, Vol. 39, pp. 521–546 (1947).

(28) Mechanical and Physical Properties of VascoJet 1000, Vanadium-Alloys Steel Co. (1959).

(29) G. Sachs, J. G. Sessler, and K. S. Grewal, "Investigation of the Effects of Stress Concentrations and the Cracking Tendency of Heat-Treated Titanium and Steel Sheet Alloys," Syracuse University, *Progress Report No. 4*, Navy BuAer Contract No. NOas 58-855-c, July 1959.

(30) E. C. Schneider, "History of Steel Rocket Motor Case Development," Pratt and Whitney Aircraft Div., United Aircraft Corp., Sept. 29, 1959.

(31) M. E. Shank, C. E. Spaeth, V. W. Cooke, and J. E. Coyne, "Solid-Fuel Rocket Chambers for Operation at 240,000 psi and Above," Parts I and II, *Metal Progress*, Nov. 1959, pp. 74–81; Dec. 1959, pp. 84–92.

(32) G. Sachs, J. G. Sessler, K. S. Grewal, and V. Weiss, "Investigation of the Effects of Stress Concentrations and the Cracking Tendency of Heat Treated Titanium and Steel Sheet Alloys," Syracuse University, *Progress Report No. 5*, on Navy BuAer Contract No. NOas 58-855c, Oct. 1959.

DISCUSSION

MR. W. F. BROWN, JR.[1] AND M. H. JONES[1] (*presented in written form*).—This paper is typical of those produced by the Research Group of Vanadium Alloys Steel Co. in that a large number of carefully conducted systematic experiments clearly define the effect of the variables and the results are presented in a lucid manner.

Room-temperature investigations of carbon content and melting practice at NASA[2] included the 5 per cent chromium steel in the form of 0.063-in. sheet. However, we used the sharp-edge notch tension test rather than the impact test to measure resistance to brittle fracture. We believe that a very sharp notch or crack is a necessary feature if sheet materials are to be evaluated regarding their tendency to catastrophic failure in service.[3]

Certain points of general agreement are immediately evident from these two investigations, namely, (1) although no hardness data are given in our paper, hardness measurements confirm that the lower-carbon alloy does exhibit the greatest secondary hardening. (2) We also find higher fracture toughness and lower sharp notch sensitivity with increasing (0.23 to 0.43 per cent) carbon level. This effect is particularly pronounced in the tensile strength range above about 175,000 psi. However, it should be noted that the authors' impact data (Fig. 4 of the paper) exhibit this effect only at strength levels above about 260,000 psi. In addition, the carbon effect in this strength range appears more clearly defined by the notch tension test than by the impact test. (3) Both the impact test (see authors' Table IV) and the sharp-edge notch test indicate that the toughness decreases rather rapidly at tensile strength levels over about 300,000 psi.

---

[1] Chief and Research Engineer, respectively, Strength of Materials Branch, Lewis Research Center, National Aeronautics and Space Administration, Cleveland, Ohio.

[2] J. L. Shannon, Jr., G. B. Espey, A. J. Repko, and W. F. Brown, Jr., "Effect of Carbon Content and Melting Practice on the Room Temperature Sharp-Edge-Notch Tensile Characteristics of H-11 Modified and 300M Sheet Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 761 (1960).

[3] "Fracture Testing of High Strength Sheet Materials: A Report of a Special ASTM Committee," ASTM BULLETIN, No. 243, Jan., 1960, p. 29, and No. 244, Feb., 1960, p. 18.

There appears, however, to be an important point of disagreement concerning the effect of vacuum melting. The authors find a considerable increase in the impact toughness of induction-vacuum-melted material. We, on the other hand, find only very small increases in the sharp-notch properties due to consumable electrode vacuum remelting. Our investigations also included a heat of induction-melted material which was not reported because the thickness (0.040 in.) was lower than for the other sheet investigated. However, the notch sensitivity of this induction-vacuum-melted alloy was at least as great as that of air-melt 0.063-in. sheet at the same carbon level. Since the notch strength would be expected to increase with decreasing thickness, the results do not indicate any benefit from induction vacuum melting. We do not, however, in any way disagree with the authors' interpretation of their data. The impact test measures a property which is more closely associated with plain strain fracturing such as is represented by a crack propagating through the thickness. It may be that had we made notch tests on round bars, vacuum-melted material would have been superior. In any event, an increase in impact toughness could indicate a corresponding increase in the average stress necessary to cause propagation of internal cracks to the sheet surface. What is needed are additional experiments on sharply notched thick specimens.

We agree with the authors that the sheet elongations may be misleading. Furthermore, we wonder what use is made of either elongation or reduction of area values in design studies that pertain to high-strength components. There is no known relation between them and the fracturing behavior in the presence of cracks.

In conclusion, we would like to ask if the authors have any explanation for the increase in toughness at the higher strength levels associated with increasing rather than decreasing carbon contents.

MR. J. C. HAMAKER (*author*).—We wish to thank Messrs. Brown and Jones for the comparative data from their study of these steels; they provide a valuable addition to the paper.

The apparent anomaly in 5 per cent chromium steel of increasing, rather than decreasing, notch toughness as the carbon level is raised from 0.20 per cent to 0.50 per cent, may be related to the alloy carbon ratio effect described by Kuo (reference (2) in the paper) and discussed in connection with the secondary hardening response under "Preliminary Heat Treating Studies" in the paper. By lowering the carbon at constant alloy content, the alloy - carbon ratio is increased. Apparently this changes the nature and/or distribution of alloy carbide precipitation at the secondary hardness peak, thereby altering the hardness response and notch toughness characteristics.

In previously reported studies of medium- and high-alloy hardenable steels, we have noted a relationship between carbon and alloy content for maximum strength capability. With a relatively low alloy content, peak strength is obtained at moderate hardness and carbon levels. With higher alloy contents, higher peak strengths are obtained at higher carbon and hardness levels. Thus, each alloy composition appears to have an optimum carbon content and heat-treated hardness level to achieve its best combination of mechanical properties. For the 5 per cent chromium steels described in the paper, the optimum levels appear to be 0.35 to 0.50 per cent carbon in the Rockwell hardness range 46 to 56, C scale.

MR. J. A. KIES.[4]—There are many uses for these steels that make the authors' criticism of decarburization quite valid, but if we focus attention on the burst tests of motor cases and small size cylinders, there are much data that tell us that a few thousandths of an inch of decarburization is good. I would not like to see the authors' results on Charpy bars extrapolated to say that decarburization is always bad.

MR. BROWN.—That is true for many low-alloy steels, but I wonder if it is true for H-11. Both those results presented by the authors and data from our own laboratory indicate that for the range of carbon contents investigated there was a decrease in the toughness with decreasing carbon content. I wonder if Mr. Kies would define more specifically the data to which he refers. It would be of interest to know the materials, types of tests, and the amount of decarburization.

MR. KIES.—The U. S. Steel Co., I believe, had some data on decarburization in H-11 in their burst chamber program, and the Pratt & Whitney Co. has been studying controlled decarburization in H-11 and other steels. I know of no case where it was found that decarburization in a thin layer of a few thousandths of an inch was bad. We should recognize that the microstructure resulting from decarburization is not the same as that obtained by heat treating a lower-carbon steel; therefore the results quoted by Mr. Brown may not prove his point. I believe that we could greatly benefit by making simple bend tests on coupons with and without decarburization. The tension surface of the specimens should be polished so that the tendency of inclusions and other flaws to start surface cracks could be noted.

As you all know, the reason why energy is absorbed in fracturing is that plastic flow occurs, and without regard to the microstructure; if plastic flow can occur to considerable extent we will find a big shear lip development. In general, the lower the yield strength is, the lower the stress at which yielding begins. We want to accommodate surface cracks. We would be glad to use glass at 500,000 or 1,000,000 psi tensile strength if it had enough plasticity to relieve surface flaws.

MR. BROWN.—I am still puzzled by the decarburization discussion and the points raised by Mr. Kies. In our tests the per cent shear followed the same trend as the fracture toughness values. In other words, we got lower per cent shear for the low-carbon levels in H-11 modified steel. However, I will admit that the structure in a decarburized surface may not correspond to that in parent metal of uniform carbon content. In this respect, the suggestion by Mr. Kies of testing bend specimens should be helpful in clarifying the problem.

MR. HAMAKER (*author's closure*).—The points raised by Messrs. Brown and Kies on decarburization are well taken. Whether decarburization of 5 per cent chromium steel improves burst characteristics of rocket cases by encouraging plastic flow, or conversely, increases notch sensitivity for poorer burst properties, may be related to its severity, that is, the surface carbon content and depth of carbon depletion in the particular case under study.

A few thousandths of *controlled* partial decarburization, having 0.30 to 0.35 per cent carbon at the surface, as in the Pratt & Whitney 5 per cent chromium steel cases mentioned by Mr. Kies, appears to be definitely beneficial. It should be remembered, however, that this decarburization is produced under very carefully controlled conditions during heat treatment, and is not related to the unavoidable, and frequently nonuniform, decarburization that occurs during the hot rolling of sheet. The successful 240,000 psi yield strength Pratt & Whitney 5 per cent chromium steel cases and the Allison D6ac cases are both made from roll ring forgings which are machined on all surfaces before final heat treatment. In addition to the obvious advantage of eliminating longitudinal welds, the use of "metallurgically clean," controlled surface conditions might contribute significantly to their success.

---

[4] Physicist, Naval Research Laboratory, Washington, D. C.

On the other hand, there have been rocket case, pressure vessel, and notch test studies in which very little attention has been given to metallurgical surface condition in the as-received material or after heat treatment. A review of the latter studies has persistently pointed to uncontrolled decarburization as a cause of substandard results where 5 per cent chromium steels were investigated. These low results with heavy decarburization would appear to be explained by the consistent relationship of low carbon to notch sensitivity observed in three independent investigations: the present, and those reported by Brown et al[2] and Klier.[5]

It should be noted that these findings for 5 per cent chromium steels do not necessarily apply to low-alloy, nonsecondary hardening steels. For example, Brown et al[2] showed, in side-by-side tests, that raising the carbon content increased the notch sensitivity of a low-alloy, 300M type steel, but reduced the notch sensitivity of the 5 per cent chromium steels. Similarly, in the U. S. Steel burst program mentioned by Mr. Kies, inadvertent heavy decarburization of the low-alloy, non-secondary hardening steels changed the results from very low stress brittle failures to fairly satisfactory burst performance. With 0.006 to 0.010 in. decarburization, all of the low-alloy steels, with the exception of the relatively low strength 4130, consistently gave low stress brittle failures. However, with 0.015 to 0.024 in. decarburization, or approximately 50 per cent of the sheet thickness, satisfactory burst characteristics were obtained. This large amount of decarburization, of course, reduced the net strength of the material by some 40,000 to 50,000 psi, so that it was no longer truly ultra-high-strength steel. Five per cent chromium steel was also included in this program, but it resisted decarburization to such an extent that only a small amount was obtained in all tests and a clear-cut comparison of the effect could not be obtained.

Summarizing the limited evidence to date, it would appear that, for the 5 per cent chromium modified H-11 steel, a small amount of partial decarburization may be beneficial, but heavy decarburization probably increases notch sensitivity. Low-alloy, non-secondary hardening steels, on the other hand, appear to show improved burst characteristics with heavy decarburization, but may require such a large amount (50 per cent of sheet thickness?) that material strength is drastically reduced, and proper surface chemistry control to the depth required may be impossible in practical hot working or heat treating operations.

---

[5] E. P. Klier, "Effect of Carbon Content on the Notch Properties of 43XX-Vanadium Modified and 5 per cent Chromium Sheet Steels," Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials, *ASTM STP No. 287*, Am. Soc. Testing Mats., p. 196 (1961).

SPIN-FRACTURE TESTS OF NICKEL-MOLYBDENUM-VANADIUM ROTOR STEELS IN THE BRITTLE FRACTURE RANGE*

By G. O. Sankey[1]

Synopsis

This paper summarizes the results of spin fracture tests of 32 test rotors taken from five generator rotor forgings. Tests were made at rotor temperatures of 80 F and 0 F. The rotors were notched at the bore to produce fracture in the brittle region, and rotor lengths were sufficient to ensure conditions near those of plane strain. As predicted by theory, it was found that the fracture stress is inversely proportional to the square root of the notch length. Fracture stress depends on the transition temperature and the ductility of the material. An improved correlation of the ratio of rotor fracture stress and ultimate strength of the material is obtained with a parameter that includes both transition temperature and tensile ductility. Prestressing at a seasoning temperature of about 270 F showed significant increase in rotor fracture stress on subsequent test at 0 F. A limited number of tests were made on the effect of notch acuity. A rotor with a notch root radius of 0.0015 in. failed at about 25 per cent lower stress than one with a notch radius of 0.008 in. In another pair of rotors with otherwise identical dimensions, the one with a notch radius of 0.001 in. failed at about 12 per cent lower stress than the one with a notch radius of 0.004 in.

List of Symbols

C = notch length of test rotor, in.,
D = outside diameter of test rotor, in.,
E = modulus of elasticity, psi,
$\mathcal{G}_c$ = fracture toughness of material,: $\frac{\pi\sigma_0^2}{\mathrm{E}}\left(\frac{C}{2}\right)$, in-lb per sq in.
h = length of the test rotor, in.,
R = outside radius of test rotor, in.,
R_n = notch root radius of test rotor, in.,
N = fracture speed of test rotor, rpm; $\omega = \frac{2\pi N}{60}$, rad per sec,
T_{50} = Charpy V-notch 50 per cent shear-fracture appearance transition temperature deg Fahr,
ΔT = ductility function, deg Fahr,
T_{pc} = precracked transition temperature, deg Fahr,
T_{eff} = effective transition temperature, $T_{50} + \Delta T$, deg Fahr,
x = index of plane strain, $\frac{h\sigma_y{}^2}{E\mathcal{G}_c}$ dimensionless,

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Mechanics Department, Westinghouse Research Laboratories, Pittsburgh, Pa.

e = elongation in 2-in. gage length, per cent,

μ = Poisson's ratio,

ρ = mass density of material, lb-sec^2 per in.^{4},

σ_0 = nominal fracture stress. Tangential stress at center of solid rotor corresponding to the fracture speed of the test rotor, $\left(\frac{3+\mu}{8}\right)\rho\omega^2R^2$, psi,

σ_0' = tangential stress at center of solid rotor corresponding to the fracture speed of the test rotor corrected for plane strain,

$$\sigma_0' = \sigma_0\left(1 + \frac{1.4}{\alpha^2}\right)^{-1/2}, \text{ psi},$$

σ_y = yield strength of material, psi (0.2 per cent offset), and

σ_u = ultimate strength of material, psi.

The main element of a modern turbine or generator rotor is an alloy steel forging. Safe designs of the single forging rotor depend on a rigorous testing and acceptance practice, and a critical part of this practice is the use of ultrasonic testing to locate and identify discontinuities (**1**).[2] Sound test rotors fail at average stresses very near the tensile strength of the material. On the other hand, rotors have failed at stresses well below the yield strength of the material (**2**, **3**, **4**). The failure surfaces in these cases exhibited little plastic deformation, and the origin of fracture was at a severe stress raiser in the body of the rotors. These failures have served to reemphasize the rotor strength problem and in particular have focused attention on the brittle behavior of the alloy steel forgings.

This investigation concerns rotor fracture strength in the brittle region as influenced by the size and location of the defect and by the mechanical properties of the material. Since it is not practical to spin full-size rotors to destruction, use is made of spin test facilities (**5**) for bursting "small" rotors of the order of 8 to 20 in. in diameter. For such tests to be meaningful, fractures of a brittle nature must be obtainable at low values of stress, comparable to service failures. Fractures in the brittle region have been possible by using "effectively notched" test rotors (**6**).

The paper summarizes results of spin fracture tests of relatively small rotors taken from nickel-molybdenum-vanadium generator rotor forgings. The test program was designed (1) to study the effect of ultimate strength, yield strength, transition temperature, and ductility of the material on the brittle fracture of rotors; (2) to establish for this class of rotor steel (nickel-molybdenum-vanadium) whether or not the relationship between fracture stress and notch length is that predicted by the Griffith-Irwin theory (**7**), particularly for the case of plane strain; and (3) to investigate the effect of notch acuity on fracture stress. It was later found desirable to determine if prestressing of rotors at about 270 F had a beneficial effect on the subsequent fracture test at lower temperature.

TEST ROTORS

Test rotors were taken from typical generator forgings or their prolongations. Rotor size was 7.75 to 20 in. in diameter and 2 to 10 in. in length. Each test rotor was notched at the bore to similate a large defect in a forging at this critical location.

---

[2] **The boldface numbers in parentheses refer to the list of references appended to this paper.**

Material:

Five nickel-molybdenum-vanadium forgings were selected for test on the basis of their differences in mechanical properties. Ultimate strength varied from 100,000 to 120,000 psi, yield strength from 75,000 to 100,000 psi, elongation (2-in. gage length) from 14 to 23 per cent and the 50 per cent shear fracture appearance transition temperature, T_{50}, from 100 to 200 F. Chemical composition was within the following range:

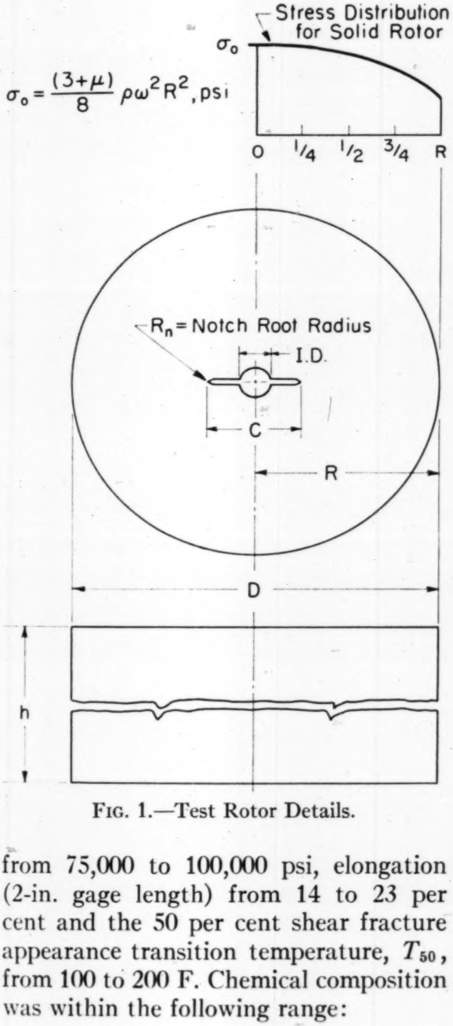

Stress Distribution for Solid Rotor
σ_o
$\sigma_o = \frac{(3+\mu)}{8} \rho \omega^2 R^2$, psi
0
1/4
1/2
3/4
R
R_n = Notch Root Radius
I.D.
C
R
D
h

FIG. 1.—Test Rotor Details.

Carbon, per cent	0.23	to 0.31
Nickel, per cent	2.5	to 2.9
Molybdenum, per cent	0.48	to 0.60
Vanadium, per cent	0.07	to 0.10
Manganese, per cent	0.55	to 0.75
Chromium, per cent	0	to 0.30
Silicon, per cent	0.18	to 0.27
Phosphorus-Sulfur, per cent	0.007	to 0.03

Notch Details:

Two radial slots were machined at the bore as shown in Fig. 1. The slots terminated at an angle of 45 deg to a prescribed root radius, R_n, at the apex. Except as noted, the notch length, C, was one quarter of the rotor diameter; the notch root radius was 0.004 in. for rotors of 7.75 in. in diameter and proportionately larger for rotors of a larger size.

Rotor Length:

Rotor lengths were sufficient to produce fracture near the conditions of plane strain. The length criterion used to determine the degree of plane strain and a method for correcting the test results to the completely plane strain case are given by Irwin (8).[3] As a practical matter one is interested in fracture of test rotors within about 15 per cent of the fracture stress corresponding to plane strain. This means that for materials with a high transition temperature of about 200 F and a high yield strength of about 100,000 psi, a rotor length of about 2 in. is required when tests are made at 0 F. For materials with a lower transition temperature of about 100 F, and a lower yield strength of about 75,000 psi, a rotor length of about 5 in. is required. When tests are made at a higher temperature, the rotors must be made longer. For example, the 2-in. and 5-in. long rotors mentioned above for tests at 0 F would have to be increased to about 3½ in. and 8 in. long, respectively, for tests at 80 F.

TEST RESULTS

Effect of Notch Length:

The effect of notch length on fracture stress was determined by a series of

---

[3] Irwin's development for plane strain is outlined in the Appendix.

TABLE I.—NOTCH LENGTH TEST RESULTS.

Nickel-Molybdenum-Vanadium Rotor Steel 4345-2	Rotor Number	Outside Diameter, in. $\left(R = \frac{D}{2}\right)$	Inside Diameter, in.	Rotor Length h, in.	Notch Root Radius, R_n, in.	Notch Length, C, in.	$\frac{C}{D}$ Ratio	Test Temperature, deg Fahr	Fracture Speed, N, rpm	Nominal Fracture Stress, $\sigma_0 = \left(\frac{3+\mu}{8}\right)\rho\omega^2R^2$
Properties at 80 F		OD	ID	h	R_n	C	$\frac{C}{D}$	deg Fahr	N	σ_0
σ_u = 120 000 psi	1...	7.75	0.687	4	0.004	1.93	0.25	80	38 520	73 800
σ_y = 100 000 psi	2...	15.5	1.375	2	0.008	3.86	0.25	5	13 650	37 200
Reduction of area = 50 per cent	3...	7.75	0.687	4	0.004	1.93	0.25	5	31 680	50 000
	4...	15.5	1.375	2	0.008	3.86	0.25	80	17 400	60 300
e = 18 per cent	5...	15.5	0.687	2	0.004	1.93	0.125	5	16 920	57 100
	6...	7.75	0.687	1	0.004	1.93	0.25	2	36 480	66 200
Transition Temperature	7...	7.75	0.687	2	0.004	1.93	0.25	2	34 080	57 800
T_{50} = 180 − 205 F	8...	7.75	0.687	3.98	0.004	1.93	0.25	−2	26 640	35 400
T_{pc} = 160 − 180 F	9...	7.75	0.687	2	0.004	1.93	0.25	−2	32 640	53 100
	10...	13.5	1.198	2	0.007	3.36	0.25	80	19 968	60 300
	11...	11.6	1.032	2	0.006	2.94	0.25	−1	20 400	46 400
	12...	20.0	1.774	2	0.010	5.0	0.25	−1	8 985	26 800
	13...	20.0	1.774	2	0.010	5.0	0.25	−1	9 540	30 200
	14...	7.75	0.356	2	0.002	1.05	0.136	1	39 200	76 500
	15...	15.5	1.375	2	0.0015	3.86	0.25	1	11 620	26 900
	16...	7.70	0.635	1.990	crack	1.95	0.24	2	33 960	56 600

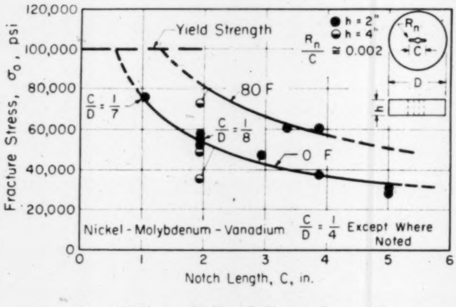


FIG. 2.—Effect of Notch Length on Fracture Strength.

tests, identified in detail in Table I. Rotors with notch lengths of 1.05, 1.93, 2.94, 3.86 and 5 in. were tested. The nominal fracture stress σ_0 is taken as the tangential stress at the center of a solid rotor corresponding to the fracture speed of the test rotor. Use of this nominal stress is similar to use of the gross section stress for plates. The tangential stress distribution for a solid rotor is shown in Fig. 1 and σ_0 is given by

$$\sigma_0 = \left(\frac{3+\mu}{8}\right)\rho\omega^2R^2$$

where:

ρ = the material mass density, lb-sec^2 per in.^{4},

ω = the rotational speed at fracture (rad per sec),

R = the outer rotor radius, in., and

μ = Poisson's ratio.

Figure 2 is a plot of the fracture stress σ_0 (from Table I) as a function of the notch length. The curve through the test values for 0 F is drawn such that σ_0 is inversely proportional to the square root of the notch length. The test values fall along this curve quite well. This is in support of the Griffith-Irwin theory. With the exception of one test (rotor 8), the scatter is about 15 per cent, which is considered reasonable. The one low result occurred on a rotor that was taken from a forging location where

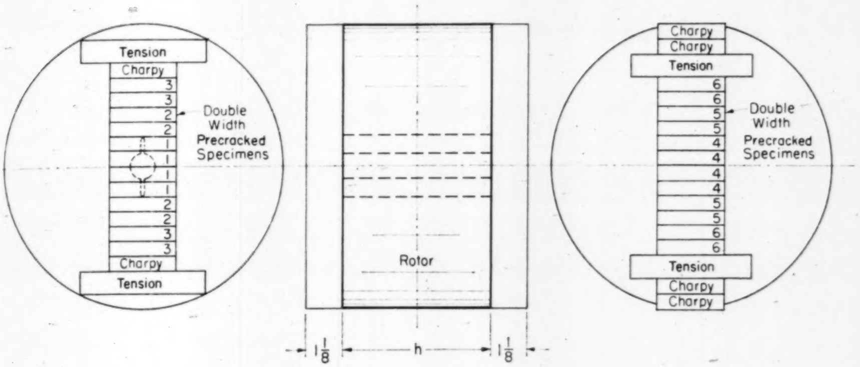

Tension
Charpy
3
3
2
2
1
1
1
1
2
2
3
3
Charpy
Tension
Double Width Precracked Specimens
Rotor
$1\frac{1}{8}$
h
$1\frac{1}{8}$
Charpy
Charpy
Tension
6
6
5
5
4
4
4
4
5
5
6
6
Tension
Charpy
Charpy
Double Width Precracked Specimens

FIG. 3.—Layout of Rotor, Tensile, Standard Charpy, and Precracked Transition Temperature Specimens.

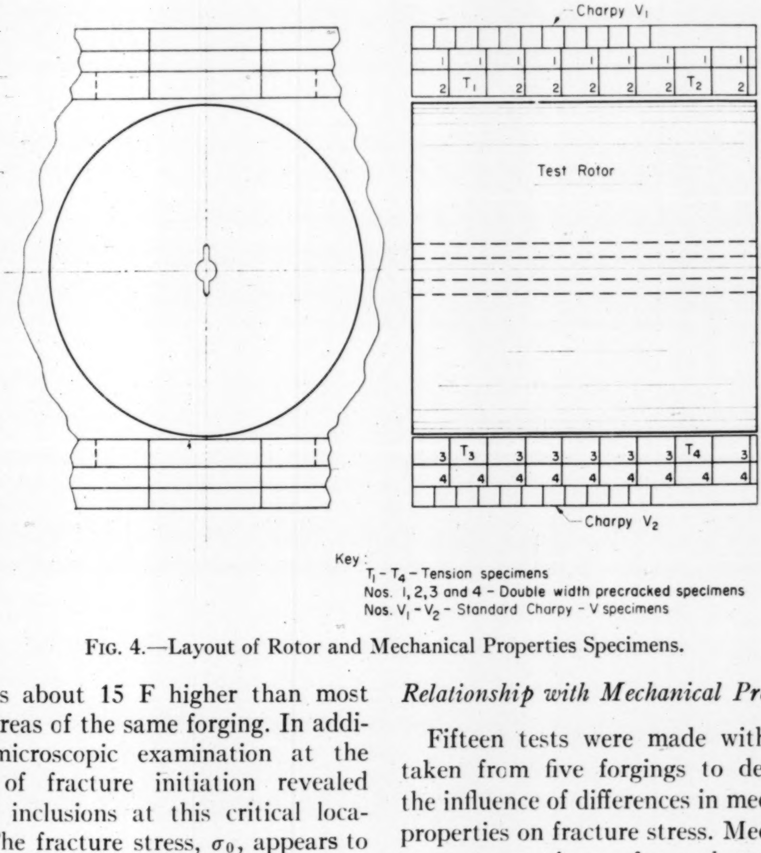

Charpy V_1
1
2
T_1
T_2
Test Rotor
T_3
T_4
3
4
Charpy V_2
Key:
$T_1 - T_4$ – Tension specimens
Nos. 1, 2, 3 and 4 – Double width precracked specimens
Nos. $V_1 - V_2$ – Standard Charpy – V specimens

FIG. 4.—Layout of Rotor and Mechanical Properties Specimens.

T_{50} was about 15 F higher than most other areas of the same forging. In addition, microscopic examination at the origin of fracture initiation revealed silicate inclusions at this critical location. The fracture stress, σ_0, appears to be inversely proportional to the square root of the notch length for the tests at 80 F also, although only three rotors were tested at this temperature.

Relationship with Mechanical Properties:

Fifteen tests were made with rotors taken from five forgings to determine the influence of differences in mechanical properties on fracture stress. Mechanical property specimens for each test rotor were taken as shown in Figs. 3 or 4. It is believed that the test specimens are taken from locations that best reflect

TABLE II.—SUMMARY OF MECHANICAL PROPERTIES AND RESULTS FROM ROTOR TESTS.

Material	Rotor Number	Material Properties Taken at Test Temperature						Outside Diameter, In. $\left(R = \frac{D}{2}\right)$	Inside Diamter, in.	Rotor Length, h, in.	Notch-Root Radius, in.	Notch Length, C, in.	$\frac{C}{D}$ Ratio	Test Temperature, deg Fahr	Fracture Speed, N, rpm	Nominal Fracture Stress, $\sigma_0 = \left(\frac{3+\mu}{8}\right)\rho\omega^2R^2$	$\mathcal{G}_c = \frac{\pi\sigma_0^2}{E}\left(\frac{C}{2}\right)$ (Irwin)	$\alpha = h\sigma_y^2/E\mathcal{G}_c$	σ_0 Correct for Plane Strain, $\sigma'_0 = \sigma_0\left(1 + \frac{1.4}{\alpha^2}\right)^{-1/2}$, psi	Yield Strength Ratio = $\frac{\sigma'_0}{\sigma_y}$	Ultimate Strength Ratio = $\frac{\sigma'_0}{\sigma_u}$	Ductility Function, deg Fahr	Effective Transition Temperature, $T_{50} + \Delta T$, deg Fahr
		Ultimate Strength, psi	Yield Strength (0.2 per cent offset), psi	Reduction of Area, per cent	Elongation, per cent	Charpy-V 50 per cent Shear Transition Temperature, deg Fahr	Charpy-V Precracked Transition Temperature, deg Fahr																
		σ_u	σ_y	RA	e	T_{50}	T_{pc}	OD	ID	h	R_n	C	$\frac{C}{D}$		N	σ_0	$\mathcal{G}_c$	α	σ'_0	$\frac{\sigma'_0}{y}$	$\frac{\sigma'_0}{u}$	ΔT	T_{eff}
Nickel-molybdenum-vanadium	17	107 900	86 900	38	18.6	172	180	7.75	0.701	4.75	0.0035	1.93	0.25	2	32 640	53 100	301	4.1	50 800	0.58	0.47	12	184
Nickel-molybdenum-vanadium 4345-2	18	121 200	103 100	53	20.3	197	188	7.75	0.687	5.25	0.0035	1.94	0.251	2	33 600	56 200	341	5.87	55 100	0.53	0.45	8	205
	19	119 900	100 000	51	19.1	185	188	7.75	0.687	7.34	0.004	1.93	0.25	79	36 240	65 500	449	5.65	64 100	0.64	0.54	10	195
	2	123 000	104 300	45.3	19.0	205	. . .	15.5	1.375	2.0	0.008	3.86	0.25	5	13 650	37 200	293	2.5	33 500	0.32	0.27	10	215
	3	124 100	110 500	48.5	19.2	180	. . .	7.75	0.687	4.0	0.004	1.93	0.25	5	31 700	50 000	266	4.83	48 500	0.44	0.39	10	190
Nickel-molybdenum-vanadium 55P226	20	117 800	96 350	27	14.7	200	185	7.75	0.687	5.0	0.004	1.93	0.25	0	28 320	39 800	170	8.5	39 800	0.41	0.34	33	233
	21	119 600	98 800	28	14.4	203	190	15.3	1.375	4.5	0.0075	3.82	0.25	−2	12 240	29 800	180	8.5	29 800	0.30	0.25	37	240
	22	120 200	100 300	25	14.2	200	205	7.75	0.587	8.37	0.004	1.93	0.25	80	31 100	48 300	246	11.8	48 300	0.48	0.40	41	241
Nickel-molybdenum-vanadium 2759	23	101 750	81 200	48	23.1	105	59	7.75	0.687	7.45	0.004	1.93	0.25	−1	37 250	69 100	508	3.33	65 200	0.80	0.64	4	109
	24	101 800	80 400	46	19.5	105	63	15.5	1.375	4.75	0.008	3.87	0.25	0	16 560	54 600	636	1.66	44 800	0.56	0.44	9	114
	25	103 750	81 000	45	20.5	118	. . .	7.75	0.687	9.960	0.004	1.93	0.25	78	37 440	70 000	509	4.43	67 700	0.84	0.65	7	125
	26	101 600	81 100	46	21.4	120	. . .	7.75	0.687	8.146	0.004	1.93	0.25	−1	33 100	54 500	311	5.93	53 500	0.66	0.53	6	126
Nickel-molybdenum-vanadium 1584	27	106 800	84 980	50	21.2	172	142	7.75	0.687	9.1	0.004	1.93	0.25	1	29 950	44 700	208	10.9	44 700	0.53	0.42	6	178
	28	106 500	84 600	49	20.7	172	140	7.75	0.687	9.1	0.004	1.93	0.25	79	35 660	63 400	418	5.35	62 100	0.73	0.58	7	179
	29	110 500	90 000	46	19.9	170	. . .	7.75	0.687	9.59	0.001	1.93	0.25	1	28 080	39 200	161	14.8	39 200	0.42	0.35	. . .	

the properties of the rotor material at the origin of fracture. The tension specimens were tested at the same temperature as that maintained in the spin tests to determine the ultimate strength, the 0.2 per cent yield strength, the reduction of area, and the elongation in a 2-in. gage length. The transition temperature T_{50} was obtained by tests of 10 to 15 standard Charpy specimens. The recently developed precracked (low-blow) transition temperature, (9) T_{pc}, was obtained from tests of 22 to 30 double width Charpy specimens. Fracture stresses σ_0 of the test rotors and the corrected values σ_0' for plane strain are shown in Table II with the corresponding mechanical properties and rotor dimensions. All other significant quantities for the test rotors are included in the table.

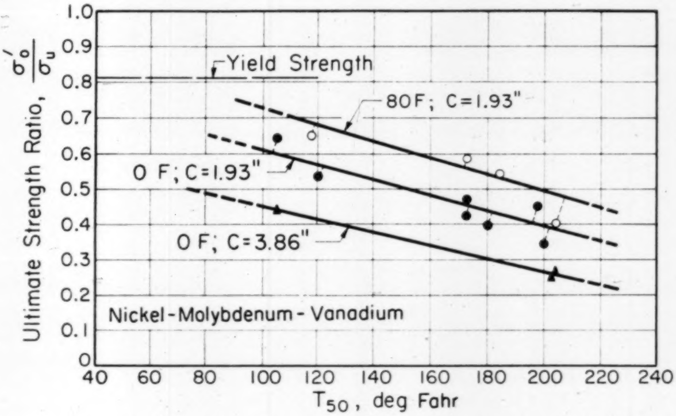


FIG. 5.—Effect of Transition Temperature T_{50} on Ultimate Strength Ratio.

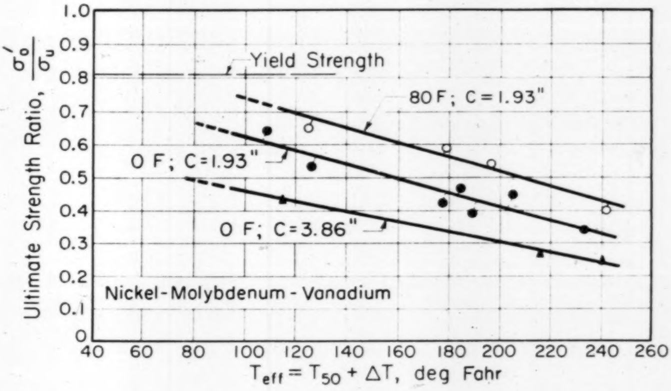


FIG. 6.—Effect of Transition Temperature T_{50} and Ductility Function ΔT on Ultimate Strength Ratio.

The ratio of rotor fracture stress corrected for plane strain over the ultimate strength of the material is shown as a function of the transition temperature T_{50} in Fig. 5. The scatter of the test values is low except at the high transition temperature region (T_{50} equal to 200 F) where the scatter is about 18 per cent. The two materials tested having this transition temperature had identical mechanical properties with the exception of the tensile ductility. Here the difference was large; that is, 19 to 20 per cent on one material and 14 to 15 per cent on the other. Test rotors taken from the material having the lower tensile ductility fractured at stress levels consistently lower than those obtained on similar rotors from the higher ductility material.

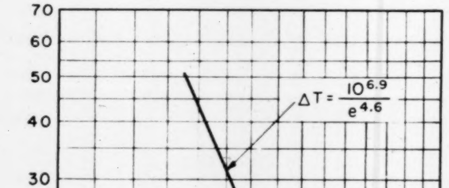


FIG. 7.—Ductility Function.

In an effort to account for the influence of tensile ductility, the data of Fig. 5 are replotted as Fig. 6 using a parameter $T_{50} + \Delta T$, where ΔT is a function of the tensile ductility. The development of the ΔT function was approached by a consistent adjustment of ΔT such that the scatter of the test points was reduced to a minimum. On this basis, ΔT is of the form $\Delta T = K/e^n$, where K and n are constants and e is the percentage elongation in a 2-in. gage length, the values of K and n are given in Fig. 7 for the material tested. The slope of the line in Fig. 7 satisfies the limited number of tests of Table II; however, this can be improved as more materials are tested. It should be mentioned that the tensile ductility is believed to be an index that reflects the ability of the material to deform rather than a direct measure of rotor fracture ductility.

In preparing curves such as Figs. 5 and 6, one might choose reduction of area as a ductility parameter and the precracked transition temperature T_{pc}; however, the scatter is more pronounced. This is due to the greater uncertainty in measurements of these quantities as compared to measurements of elongation and T_{50}.

Effect of Prestress at Seasoning Temperatures:

Generator rotors are normally seasoned at a temperature in the range of about 200 to 270 F, and it was thought possible that yielding at stress raisers during this operation would improve the fracture strength of rotors when subsequently operated at a lower temperature. Wessel (**10**) has shown that such a beneficial effect is obtained with flat tensile plates (essentially plane stress). In order to investigate the effect with rotors (plane strain), four tests were made. Notch root radius on these test rotors was reduced to 0.001 in. to more nearly approximate conditions on actual rotors containing cracks.

The test results are given in Table III where the prestress is that at the center of a solid rotor at the prestress speed. These results are plotted in Fig. 8 where the fracture stress at 0 F is shown as a function of the prestress. The test points indicate an increase in fracture stress

TABLE III.—PRESTRESS TEST RESULTS.

Material	Rotor Number	Material Properties at 0 F				T_{50} Transition Temperature, deg Fahr	Outside Diameter, in	Notch Length, in.	Notch Root Radius, in.	Rotor Length, in.	Prestress Test Parameters			Fracture Test				
		Ultimate Strength, psi	Yield Strength, psi	Reduction of Area, per cent	Elongation, per cent						Temperature, deg Fahr	Duration of Test, hr	Prestress $\left(\frac{3+\mu}{8}\right)$ $\rho\omega^2R^2$	Temperature, deg Fahr	Speed, rpm	Fracture Stress, $\sigma_0 = \left(\frac{3+\mu}{8}\right)$ $\rho\omega^2R^2$, psi	Plane Strain Index, α	Fracture Toughness, in-lb per sq in.
		σ_u	σ_y	RA	e	T_{50}	OD	c	R_n	h		hr	psi		N	σ_0	α	$\mathcal{G}_c$
Nickel-molybdenum-vanadium 1584	29	110 500	90 000	46	19.9	170	7.75	1.93	0.001	9.59	. . .	. . .	. . .	1	28 080	39 200	14.8	161
	30	107 700	87 150	47	20.0	170	7.75	1.93	0.001	9.59	273	7	60 700	0	31 380	48 900	9.5	250
	31	107 170	86 400	50	20.4	170	7.75	1.93	0.001	9.60	273	7	54 100	−1	32 000	51 000	9.08	272
	32	109 200	88 200	49	21.1	173	7.75	1.93	0.001	9.38	273	7	65 200	0	32 760	53 400	8.45	298

with increase in prestress. Wessel's data for flat tensile plates taken from a similar material are shown also. The indicated improvement for rotors is approximately one half of that for plates.

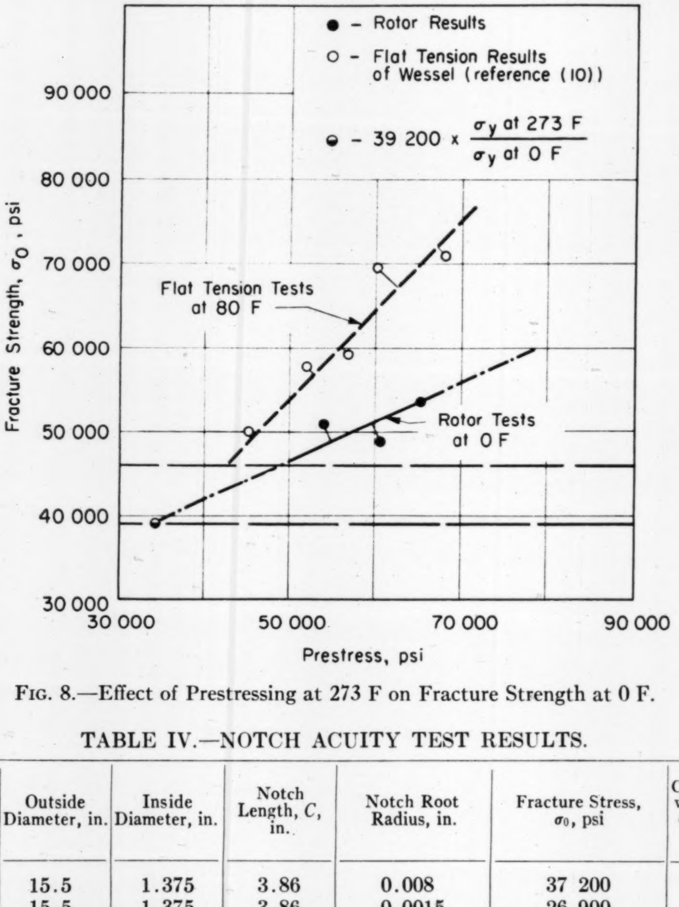


FIG. 8.—Effect of Prestressing at 273 F on Fracture Strength at 0 F.

TABLE IV.—NOTCH ACUITY TEST RESULTS.

Rotor Number	Outside Diameter, in.	Inside Diameter, in.	Notch Length, C, in.	Notch Root Radius, in.	Fracture Stress, σ_0, psi	Change in Stress with Reduction of Radius, per cent
2..........	15.5	1.375	3.86	0.008	37 200	28
15..........	15.5	1.375	3.86	0.0015	26 900	
7..........	7.75	0.687	1.93	0.004	57 800⎫ 55 450	...
9..........	7.75	0.687	1.93	0.004	53 100⎭	
16..........	7.70	0.635	1.95	crack (grain size)	56 600	
27..........	7.75	0.687	1.93	0.004	44 700	12
29..........	7.75	0.687	1.93	0.001	39 200	

The smaller improvement of fracture strength indicated for rotors as compared to plates is attributed to the higher degree of plane strain. This suggests that for complete plane strain the improvement in fracture stress would probably be reduced further. No improvement in fracture stress would be expected until the prestress exceeded the product of the fracture stress and the ratio of the material yield strengths at the prestress and test temperatures. This point shown in Fig. 8 determines, at least approximately, the slope of the curve. The improvement in fracture stress of the test rotors with increasing prestress is estimated to end at about 80,000 psi.

Effect of Notch Root Radius:

Some idea of the effect of notch root radius on fracture strength can be obtained by comparing certain of the rotor tests as relisted in Table IV. In the cases of machined notches, the effect of smaller notch root radius appears to be significant. Rotor No. 15 with 0.0015-in. notch radius, R_n, had a fracture stress 28 per cent lower than rotor No. 2 where R_n was 0.008 in. Similarly, rotor No. 29 with $R_n = 0.001$ in. had a fracture stress 12 per cent lower than rotor No. 27 where $R_n = 0.004$ in. Rotors No. 7 and 9 with $R_n = 0.004$ in. are compared with rotor No. 16 where rotor No. 16 had a crack induced by an explosive charge in a bore slot. It was somewhat surprising that the cracked rotor failed at about the same stress as the rotors with machined notches; however, a possible explanation may be that the explosive charge produced a blunt-ended crack.

SUMMARY

Spin fracture experiments of effectively notched test rotors have evaluated the brittle behavior characteristics of nickel-molybdenum-vanadium rotor steel. The test program was designed to study rotor fracture strength as influenced by notch length and acuity and the mechanical properties of the material. Rotor length was made sufficiently long to ensure conditions near those of plane strain.

As predicted by the Griffith-Irwin theory, it was found that the fracture stress is inversely proportional to the square root of the notch length.

An improved correlation of the ratio of rotor fracture stress and ultimate strength of the material was obtained with a parameter that includes both transition temperature and ductility.

A limited number of tests were made to study the effect of notch acuity on rotor fracture stress. For the case of machined notches, reducing notch root radius from 0.008 to 0.0015 in. reduced the fracture stress by 28 per cent. In another pair of otherwise similar rotors, a reduction of 12 per cent was obtained when the notch root radius was reduced from 0.004 to 0.001 in.

Prestressing of rotors at an elevated temperature of about 270 F had a beneficial effect on subsequent tests at 0 F. The improvement in fracture strength for rotors is about one half of that previously determined for plates.

Acknowledgment:

The spin-test program was carried out under the direction of R. E. Peterson. His advice and assistance are gratefully acknowledged. Thanks are due A. C. Hagg, M. J. Manjoine, E. T. Wessel, J. H. Bitzer, and R. R. Hovan for help in the test program. The interest and support of R. A. Baudry and R. L. Cloud of the East Pittsburgh Works and the help of J. Myers and F. Cassel of Materials Engineering are appreciated. The cooperation of J. W. Jones of duPont Eastern Laboratories in producing a rotor with a crack notch is acknowledged.

APPENDIX

The fracture stress for plane strain σ_0' can be related to the test value of the fracture stress σ_0 by

$$\sigma_0' = \sigma_0 \left(1 + \frac{1.4}{\alpha^2}\right)^{-1/2}$$

where α is an index that is related to the degree of plane strain (Irwin (**8**)). Our tests support this relation where α is about 1.5 or greater.

The index α is given by

$$\alpha = \frac{h\sigma_y^2}{E\mathcal{G}_c}$$

where:

h = the specimen thickness (rotor length), in.,

σ_y = the yield strength of material, psi,

E = Young's modulus, psi, and

$\mathcal{G}_c$ = the fracture toughness.

The fracture toughness $\mathcal{G}_c$ is determined from the rotor tests and is given by

$$\mathcal{G}_c = \frac{\pi\sigma_0^2}{E}\left(\frac{C}{2}\right) \text{ (in-lb per sq in.)}$$

where C is the notch length, in., and σ_0 is interpreted as the stress at the center of a solid rotor at the fracture speed of the test rotor. For rotors and for steel material, σ_0 is given by

$$\sigma_0 = 0.332 \times 10^3 \left(\frac{N}{10^4}\right)^2 R^2$$

where N is the fracture speed, rpm, and R is the outer rotor radius, in.

REFERENCES

(**1**) N. L. Mochel, R. E. Peterson, J. D. Conrad, and D. W. Gunther, "Large Rotor Forgings for Turbine and Generators," *Transactions*, Am. Soc. Mechanical Engrs., pp. 1585–1601 (1956).

(**2**) H. D. Emmert, "Investigation of Large Steam-Turbine Spindle," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 78, pp. 1547–1565 (1956).

(**3**) C. Schabtach, E. L. Fogleman, A. W. Rankin, and D. H. Winne, "Report of the Investigation of Two Generator Rotor Fractures," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 78, pp. 1567–1585 (1956).

(**4**) D. R. DeForest, L. P. Grobel, C. Schabtach, and B. R. Seguin, "Investigation of the Generator Rotor Burst at the Pittsburgh Station of the Pacific Gas and Electric Co.," Power Division Conference, Am. Soc. Mechanical Engrs., Allentown, Pa., Oct. 21–23, 1957.

(**5**) A. C. Hagg, B. Cametti, and G. O. Sankey "A High Speed, High Temperature Precision Testing Machine for Gas Turbine Disk Research," *Proceedings*, Soc. Experimental Stress Analysis, Vol. VIII, No. 2, pp. 17–28 (1951).

(**6**) D. H. Winne and B. M. Wundt, "Application of the Griffith-Irwin Theory of Crack Propagation to the Bursting Behavior of Disks, Including Analytical and Experimental Studies," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 80 (1958).

(**7**) G. R. Irwin, J. A. Kies, and H. L. Smith, "Fracture Strengths Relative to Onset and Arrest of Crack Propagation," *NRL Report 5222*, Nat. Research Laboratory, Nov., 1958.

(**8**) G. R. Irwin "Fracture Mode Transition for a Crack Traversing a Plate," Metals Engineering Conference, Am. Soc. Mechanical Engrs., Albany, N. Y., April 29–May 1, 1959.

(**9**) G. M. Orner and C. E. Hartbower, "Effect of Specimen Geometry on Charpy Low-Blow Transition Temperature," *Welding Journal*, Vol. 36, Dec., 1957, pp. 521s–527s.

(**10**) E. T. Wessel and R. A. Baudry, Discussion of a paper by M. Gensamer at Conference on Fracture in Engineering Materials, Troy, N. Y., Am. Soc. Mechanical Engrs., Aug., 1959.

DISCUSSION

MESSRS. A. J. BROTHERS[1] AND D. H. WINNE[1] (*presented in written form*).—We wish to welcome this addition to the literature describing the brittle fracture of notched disks. The effects of disk length, notch acuity, and mechanical properties on the bursting behavior of model rotors are very pertinent to the engineering design of full-scale turbine and generator rotors.

There are some aspects of the paper that deserve comment. For the particular disk geometries used, the author's simplified expression for $\mathcal{G}_c$, the fracture toughness, gives results that are in close agreement with those obtained from the equation derived by Winne and Wundt.[2] However, one should also consider the case in which the ratio of bore diameter to notch length is closer to unity. For this condition, the author's equation would yield results significantly different from those obtained from the Winne and Wundt relationship.

The author's determination of the effect of notch acuity on disk burst behavior confirms the results of Lubahn and Yukawa[3] and Wundt[4] for large notched bend bars. In the July, 1960 issue of *Metals Progress*, Messrs. Newhouse and Wundt demonstrated the effectiveness of a brittle nitride layer as a means of obtaining a crack acuity approaching that of a natural crack.

It is of interest to note the author's observation of the disturbing effect of tensile ductility on the relationship between disk bursting tests and the Charpy fracture appearance transition temperature (T_{50}). DeForest, Newhouse, and Seguin[5] reported a similar ductility effect in 1957. They found it possible to account effectively for differences in ductility and strength level by using the tensile fracture strength. This stress combines both strength and ductility values into a single property of physical significance.

To determine if the method used by DeForest et al were applicable to the data in this paper, the nominal fracture stress σ_0, for a number of disks have been corrected to a common tensile fracture stress. Since fracture stresses were not reported by the author, it was necessary to estimate these values from the reported ultimate strengths and reductions of area. This was done using the data of the accompanying Fig. 9 which show that for a large number of tests of rotor steels there is a rather close relationship between tensile strength, reduction of area, and tensile fracture stress.

The corrected nominal fracture stresses are shown in the accompanying Table V. Correction to a common tensile fracture stress almost completely eliminates the difference in fracture stress between otherwise similar disks of different ductility levels.

---

[1] General Electric Co., Large Steam-Turbine Generator Department, Schenectady, N. Y.

[2] D. H. Winne and B. M. Wundt, "Application of the Griffith-Irwin Theory of Crack Propagation to the Bursting Behavior of Disks, Including Analytical and Experimental Studies," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 80 (1958).

[3] J. D. Lubahn and S. Yukawa, "Size Effects in Slow Notch-Bend Tests of a Nickel-Molybdenum-Vanadium Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 661 (1958).

[4] B. M. Wundt, "A Unified Interpretation of Room-Temperature Strength of Notched Specimens as Influenced by Their Size," *ASME Paper No. 59-MET-9*, Am. Soc. Mechanical Engrs., May 3, 1959.

[5] D. R. DeForest, D. L. Newhouse, and B. R. Seguin, "Progress in the Development of Steam Turbine-Generator Rotor Materials," *ASME Paper No. 57-A-280*, Am. Soc. Mechanical Engrs., Dec. 6, 1957.

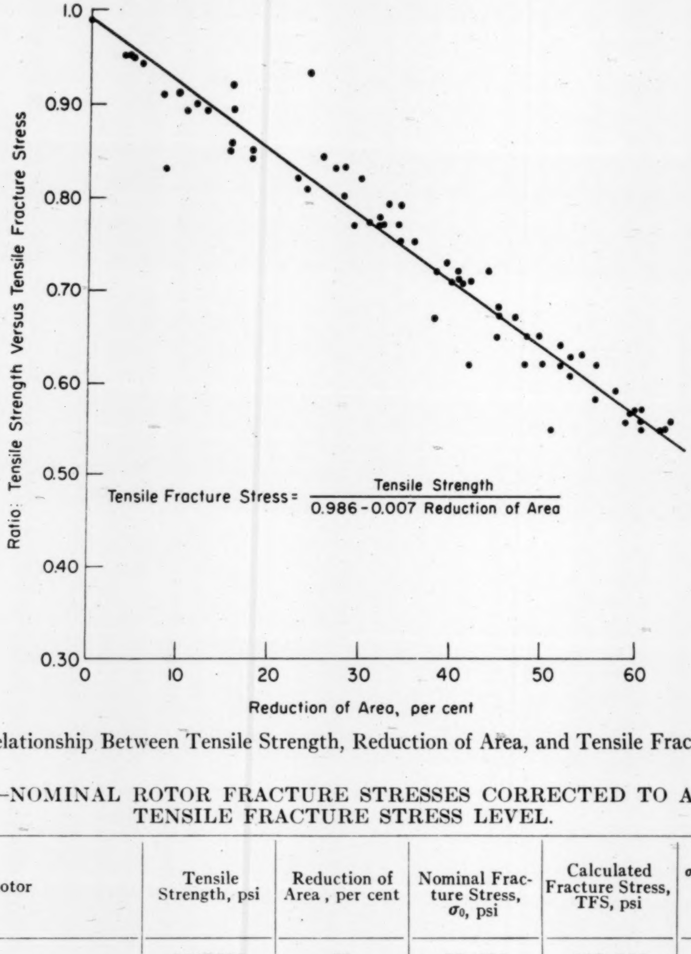


FIG. 9.—Relationship Between Tensile Strength, Reduction of Area, and Tensile Fracture Stress.

TABLE V.—NOMINAL ROTOR FRACTURE STRESSES CORRECTED TO A COMMON TENSILE FRACTURE STRESS LEVEL.

Rotor	Tensile Strength, psi	Reduction of Area, per cent	Nominal Fracture Stress, σ_0, psi	Calculated Fracture Stress, TFS, psi	σ_0 Corrected to 190 000 psi. TFS
No. 18	121 000	53	56 200	198 000	54 000
No. 20	118 000	27	39 800	151 000	50 100
No. 19	120 000	51	65 500	192 000	64 900
No. 22	120 000	25	48 300	148 000	62 000
No. 2	123 000	45	37 200	184 000	38 400
No. 21	120 000	28	29 800	152 000	37 200

In closing, we would like to comment on the feasibility of full-scale rotor burst tests. Figure 10 shows three of six generator test rotors prior to spinning in our rotor overspeed and bursting facility near Schenectady. These are full-diameter generator fields containing slots filled with field conductors as in normal operation; the body length is 6 ft. Figure 11 shows the facility. Of the six rotors tested here, three have been notched along the entire bore to duplicate the notches used

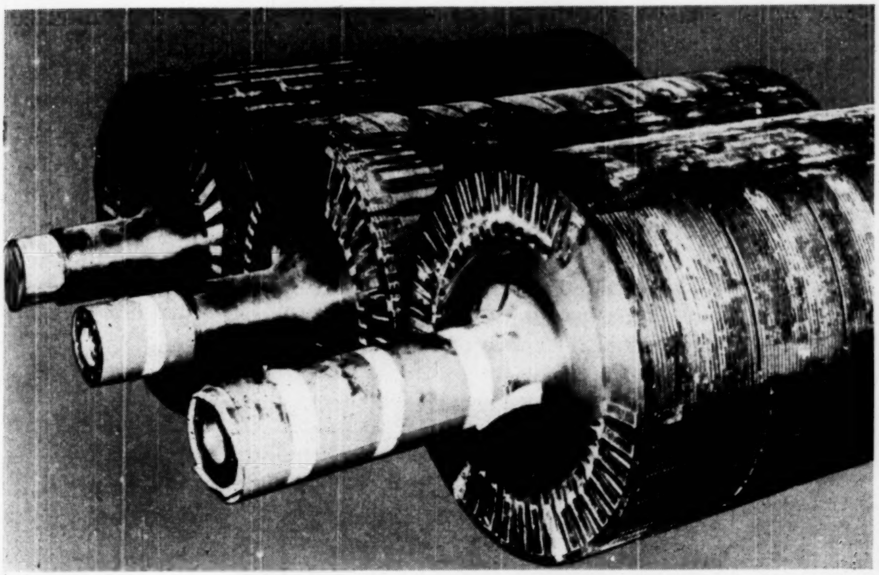


FIG. 10.—Generator Field Test Rotors.

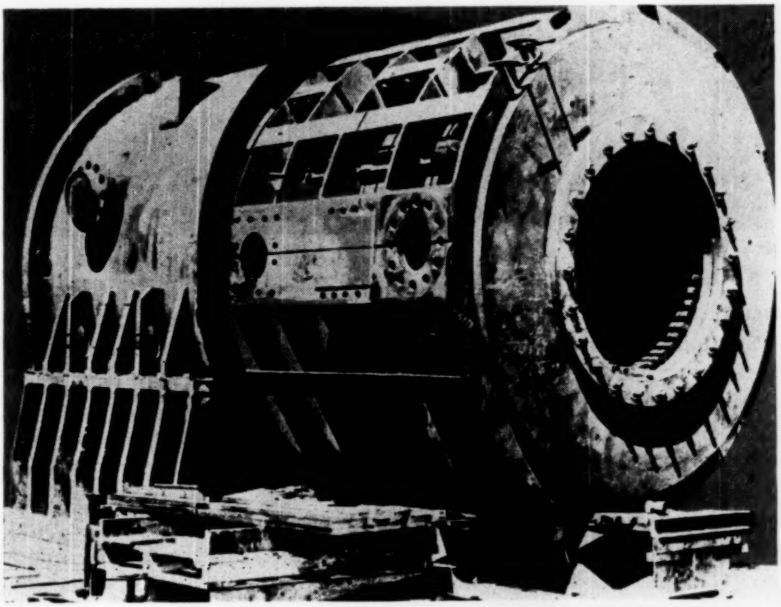


FIG. 11.—General Electric Co.'s Rotor Bursting and Overspeed Facility.

in the bore of smaller spin disks. The fracture surface of one of these very long test rotors is shown in Fig. 12. This test and others which have been described in the August and September, 1959, issues of *Metals Progress* have contributed to increasing our understanding of the variables which effect rotor bursting behavior.[6]

MR. GEORGE R. IRWIN.[7]—This is more of a comment than a question.

For those interested in niceness of experimental techniques, the air support for the spinning equipment deserves a compliment as a good job. Difficult experimental troubles were overcome in order to do the tests in a nice way.

Having recognized the excellent experimental technique, let us bear in mind that the field of stress is essentially a static field and there are differences introduced in the test results from different amounts of slow growth as affected by notch sharpness. In a truly static test method in which the test specimen is stationary, for example, a sharply notched round tension bar, the amount of slow growth could be measured by ink staining and this variable could be taken out in the calculation of test results. Both in the spin tests at the General Electric Co. and the spin tests at Westinghouse Electric Corp., I am hopeful that attention will be paid to substitution for these, impressive as they are, types of tests of somewhat more convenient purely static tests in which the slow growth of the crack can also be studied.

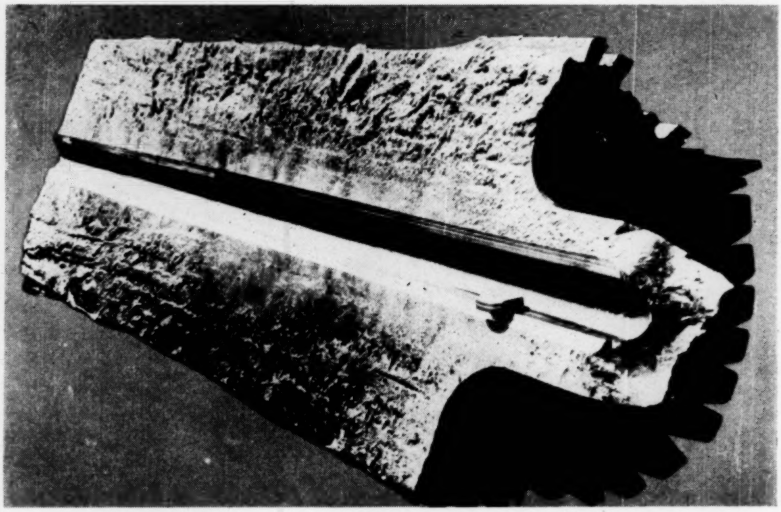


FIG. 12.—Fracture Surface of Burst Test Rotor.

MR. G. O. SANKEY (*author's closure*).—The author wishes to express his appreciation for the interesting discussions presented with reference to the paper.

Messrs. Brothers and Winne point out that the Griffith-Irwin equation used for calculating $\mathcal{G}_c$ without reference to the rotor bore is valid provided the slot lengths extending from the bore are relatively long compared to the bore dimensions. For designs where the slot lengths on each side of the bore are equal to or less than the bore radius, the modified expression of Winne and Wundt should be used.

---

[6] E. E. Thum, "Bigger, Better and Sounder Forgings," *Metals Progress*, Aug. and Sept., 1959.

[7] Superintendent, Mechanics Division, U. S. Naval Research Laboratory, Washington, D. C.

The influence of notch acuity on the fracture strength of rotors is, of course, very important. A brittle nitrided layer through which a crack can develop at low values of strain bears comment. This suggestion would appear to offer an excellent method of obtaining test rotors having defects simulating crack conditions, assuming no metallurgical changes occur during the nitriding operation.

It is reassuring to learn that DeForest, Newhouse, and Seguin have found a similar tensile ductility effect on the relationship between fracture strength and the mechanical properties of the material. The important comparison shown in Table V is that the ratio of the nominal fracture stresses σ_0 from disk tests is nearly the same as the ratio of the calculated true fracture stresses (TFS) from mechanical property tests; thus, two materials with similar tensile strength will have different values of fracture strength depending upon their ductilities. With equal strain hardening coefficients the material with the higher ductility will develop the greater fracture strength.

As Mr. Irwin points out, the spin test is a convenient static test to apply large forces to large sections. At Westinghouse considerable research is under way on other static tests which will allow a study of the slow growth of cracks.

TEMPERATURE DEPENDENCE OF THE ELASTIC MODULI OF SEVERAL STAINLESS STEELS*

By F. Garofalo[1]

Synopsis

The elastic moduli of two austenitic stainless steels, one a chromium-manganese-nitrogen (Cr-Mn-N) steel and the other a chromium-manganese-molybdenum-vanadium-nitrogen (Cr-Mn-Mo-V-N) steel, and a ferritic stainless steel, modified type 422, have been determined at temperatures between 80 and 1000 F. The elastic moduli and the temperature dependence of the austenitic steels differ in some respects from those found for the standard AISI 300 grades. A measurable difference in behavior is also found for the modified type 422 steel when compared to a type 410 ferritic stainless steel.

Although it is generally accepted that the elastic moduli of polycrystalline metals and alloys are insensitive to microstructural changes, this is not necessarily so. Usually, no really large changes in the elastic moduli are found, particularly at room temperature, in cases where changes in microstructure are observed. However, an effect of grain size has been observed (**1,2**),[2] for example, at sufficiently high temperatures where grain boundary shearing becomes of importance. In addition, at both room temperature and at elevated temperatures the elastic moduli are affected measurably by alloying elements which, as far as is known, are retained in solid solution (**3-6**).

In the present study, the temperature dependence of the elastic moduli and of Poisson's ratio has been determined for two commercial austenitic stainless steels, one a chromium-manganese-nitrogen (Cr-Mn-N) steel and the other a chromium - manganese - molybdenum-vanadium - nitrogen (Cr-Mn-Mo-V-N) steel, which differ significantly in composition from the standard AISI 300 series austenitic stainless steel, and of a modified type 422 ferritic stainless steel.

One or both of the elastic moduli of the two austenitic steels differ from those of standard austenitic stainless grades at all temperatures included in this study, 80 to 1000 F. In comparing the results obtained for the Cr-Mn-N steel with those of a type 304 austenitic stainless steel, it is found that Young's modulus, *E*, is greater for the Cr-Mn-N steel at room temperature. The shear modulus, *G*, is found to be slightly greater for one specimen, but essentially the same for a second specimen. For the cases where the moduli for the two grades differ, the difference diminishes as the temperature is increased over the range examined—that is, 80 to 1000 F. Except near room temperature, the values of Poisson's ratio are equivalent for both steels.

In comparing the behavior of the

---

* Presented at the Sixty-third Annual Meeting of the Society, June 25–July 1, 1960.

[1] Edgar C. Bain Laboratory for Fundamental Research, U. S. Steel Corp., Research Center, Monroeville, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Cr-Mn-Mo-V-N steel with that of type 316 steel, which in itself behaves similarly to other grades of the 300 series, the Cr-Mn-Mo-V-N steel exhibits higher values of Young's modulus. Good agreement is found, however, in the shear modulus. Poisson's ratio is appreciably less for the type 316 steel. A similar behavior is found when comparing the results found for the modified type 422 steel and those obtained for the type 410 steel.

TABLE I.—COMPOSITION OF STEELS.

Type	Weight, per cent							
	Carbon	Manganese	Silicon	Nickel	Chromium	Molybdenum	Vanadium	Nitrogen
Cr-Mn-V[a]	0.10	15.00	0.50	...	17.00	...	...	0.40
AISI 304[b]	0.06	0.38	0.39	9.56	18.71	...	...	0.043
Cr-Mn-Mo-V-N[a]	0.10	15.00	0.50	5.00	17.00	2.00	0.75	0.35
AISI 316[b]	0.07	1.88	0.48	9.84	17.36	2.55	...	0.048
Modified AISI 422[a]	0.25	0.60	0.60	...	12.00	1.00	0.30	0.03
AISI 410[b]	0.08	0.47	0.22	0.14	12.08	0.03	...	0.03

[a] Nominal composition.
[b] Actual composition.

MATERIALS AND TEST PROCEDURES

The composition of the three steels tested and the composition of types 304, 316 and 410 steels with which they are compared are given in Table I. The Cr-Mn-N steel was heat treated by water quenching from 2000 F. The Cr-Mn-Mo-V-N steel was solution treated at 2100 F and aged at 1300 F for 10 hr followed by air cooling. The microstructure of both steels is shown in Fig. 1. In addition to a slight difference in grain size, extensive precipitation is found in the Cr-Mn-Mo-V-N steel. The modified type 422 steel was heat treated by austenitizing at 1850 F for 15 min, air cooling and tempering at 900 F for 4 hr. The heat treatment of types 304, 316 and 410 steels has been reported previously (3).

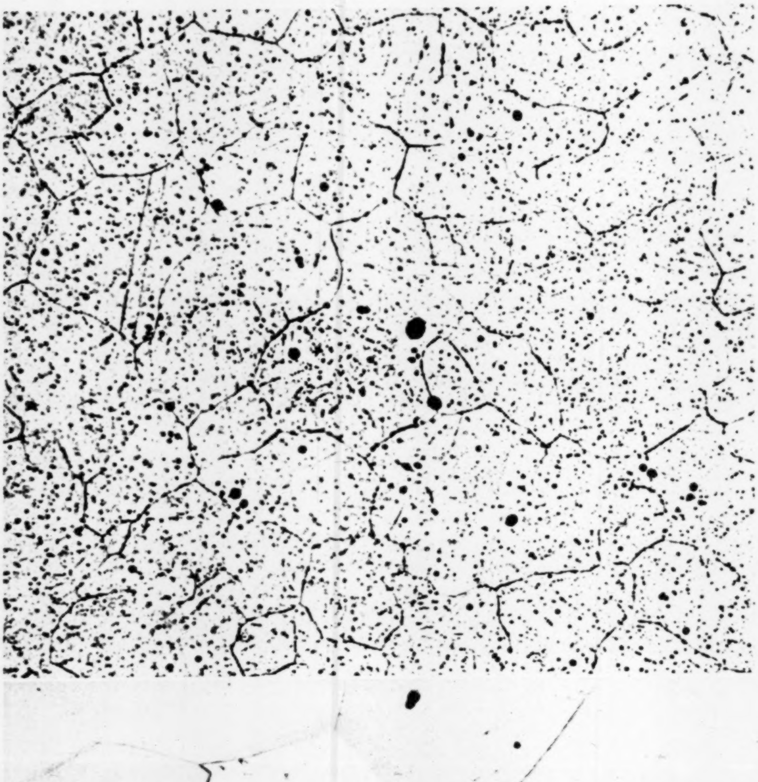
Two specimens of each steel were tested. The test procedure used in determining Young's modulus, E, and the shear modulus, G, has been described previously (3). These moduli are determined statically by subjecting a cylindrical beam, 21 in. long and 0.3 to 0.4 in. in diameter, to simultaneous bending and twisting moments. The deflections produced by both moments are magnified by an optical lever system and recorded. Three tests were made on each specimen at each temperature. The test temperatures in the present study covered a range between 80 and 1000 F. In both bending and twisting, the maximum stress reached within the gage section did not exceed 4000 psi.

TEST RESULTS

The test results for Young's modulus, E, and the shear modulus, G, between 80 and 1000 F for the Cr-Mn-N steel are given in Table II and shown graphically in Fig. 2. Each value given in Table II and each point shown in Fig. 2 represents the average of three tests. The values of Poisson's ratio, μ, are calculated from the relation

$$\mu = \frac{E}{2G} - 1 \qquad (1)$$

In Fig. 2, the test results are compared with similar data obtained previously



(*a*) (*b*)

FIG. 1.—Microconstruction of (*a*) Chromium-Manganese-Nitrogen Steel and (*b*) Chromium-Manganese-Molybdenum-Vanadium-Nitrogen Steel. Picric - Hydrochloric Reagent (× 500).

(3) on a type 304 austenitic stainless steel. Both specimens of the Cr-Mn-N steel and especially specimen No. 2, exhibit higher E values than the type 304 steel at all temperatures studied. However, as the temperature is increased, the difference decreases, being small at 1000 F.

TABLE II.—ELASTIC MODULI, E, G, AND μ DETERMINED FOR A CHROMIUM-MANGANESE-NITROGEN STEEL AT TEMPERATURES BETWEEN 80 AND 1000 F.

Temperature, deg Fahr	Specimen 1			Specimen 2		
	E, psi	G, psi	μ	E, psi	G, psi	μ
80	29.9×10^6	11.0×10^6	0.35	31.7×10^6	11.7×10^6	0.35
200	29.4	11.0	0.34	30.5	11.5	0.32
400	27.3	10.3	0.33	29.0	10.8	0.34
600	25.8	9.7	0.32	26.7	10.5	0.27
800	24.3	9.1	0.33	25.2	9.8	0.29
1000	23.1	8.6	0.33	23.2	9.0	0.28

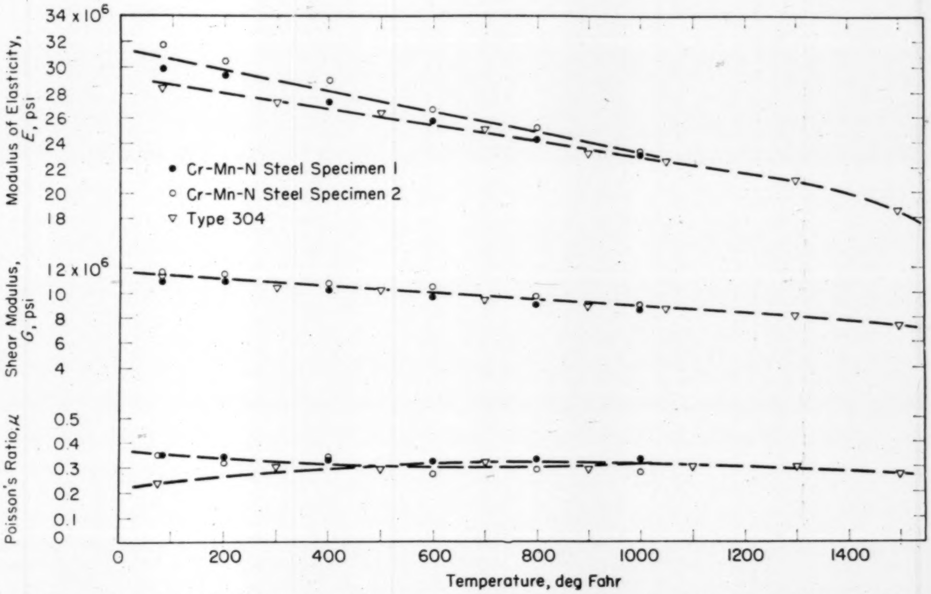


FIG. 2.—Variation of E, G, and μ with Temperature for the Chromium-Manganese-Nitrogen Steel and Type 304 Austenitic Stainless Steel.

The reason for the difference found in E between the two specimens of this steel is not known. A similar difference is also found in the G values. The shear modulus values for specimen No. 2 are slightly greater than, and those for specimen No. 1 are about the same as, those for type 304 austenitic stainless steel. The difference in the elastic moduli for the two specimens at 80 and 400 F is greater than the variation found between any two specimens for a large number of ferritic and stainless steels (3). The variability for each specimen.

that is, within three tests, is the same as that found previously for many steels. The differences found here cannot be attributed to microstructural changes since the microstructure of the two specimens is found to be identical.

TABLE III.—ELASTIC MODULI, *E*, *G*, AND μ DETERMINED FOR A CHROMIUM-MANGANESE-MOLYBDENUM-VANADIUM-NITROGEN STEEL AT TEMPERATURES BETWEEN 80 AND 1000 F.

Temperature, deg Fahr	Specimen 1			Specimen 2		
	E, psi	*G*, psi	μ	*E*. psi	*G*, psi	μ
80........	31.3 × 10^6	11.0 × 10^6	0.42	30.5 × 10^6	11.2 × 10^6	0.36
200........	31.2	11.0	0.42	30.2	11.1	0.36
400........	29.8	10.2	0.46	29.0	10.5	0.38
600........	28.3	9.5	0.49	28.7	9.9	0.45
800........	26.9	9.2	0.46	27.0	9.4	0.44
1000........	25.7	8.7	0.48	26.5	8.9	0.49

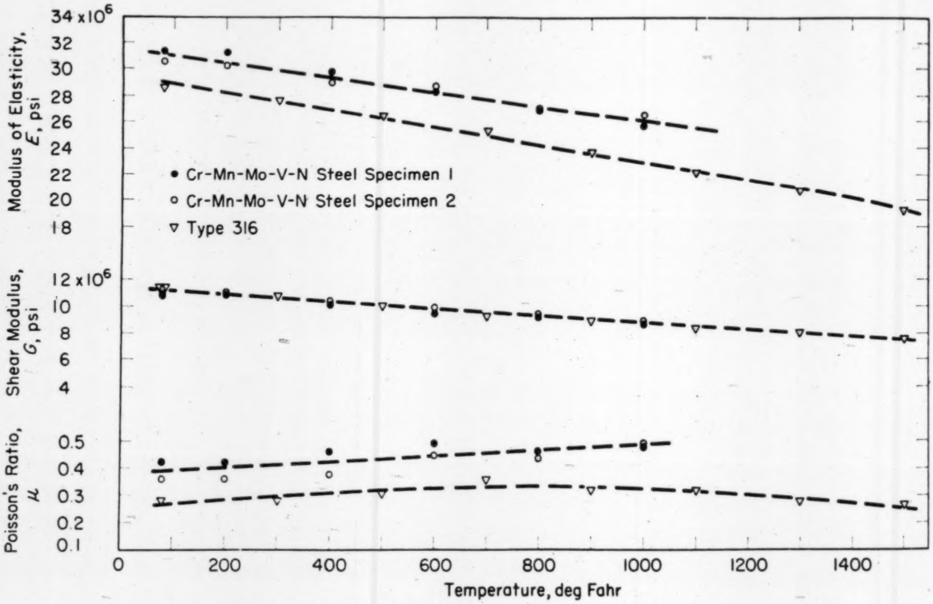


FIG. 3.—Variation of *E*, *G*, and μ with Temperature for the Chromium-Manganese-Molybdenum-Vanadium-Nitrogen Steel and Type 316 Austenitic Stainless Steel.

Except at room temperature, the Poisson's ratio values of the two specimens of the Cr-Mn-N steel are similar to those of the type 304 steel. At room temperature, Poisson's ratio for type 304 steel is lower, the difference being just slightly greater than the range in μ of 0.1 established previously for two specimens of the same material (3).

For the Cr-Mn-Mo-V-N steel the test results are given in Table III and are shown graphically in Fig. 3 where *E*, *G*, and μ for this material and for type 316 austenitic stainless steel are compared. Type 316 steel is used for this comparison[3] because, like the Cr-Mn-

---

[3] Actually, the elastic moduli for types 304 and 316 steels are very nearly the same.

Mo-V-N steel it contains normally 2 per cent molybdenum. For these two grades, E and μ differ but G does not. Young's modulus, E, is appreciably less at room temperature for the type 316 steel and the difference increases somewhat with increasing temperatures. The shear modulus, G, remains essentially the same for the two steels. Poisson's ratio is less at room temperature for type 316 steel, and the difference becomes even greater at higher temperatures.

TABLE IV.—ELASTIC MODULI, E, G, AND μ DETERMINED FOR A MODIFIED TYPE 422 STAINLESS STEEL AT TEMPERATURES BETWEEN 80 AND 1000 F.

Temperature, deg Fahr	Specimen 1			Specimen 2		
	E, psi	G, psi	μ	E, psi	G, psi	μ
80	32.0×10^6	11.7×10^6	0.36	31.5×10^6	12.1×10^6	0.30
200	31.7	11.7	0.35	31.2	11.5	0.35
400	30.6	11.0	0.39	30.0	11.3	0.33
600	29.1	10.4	0.39	29.3	10.5	0.40
800	28.1	10.0	0.41	28.0	9.9	0.41
1000	25.3	9.2	0.38	25.5	9.1	0.39

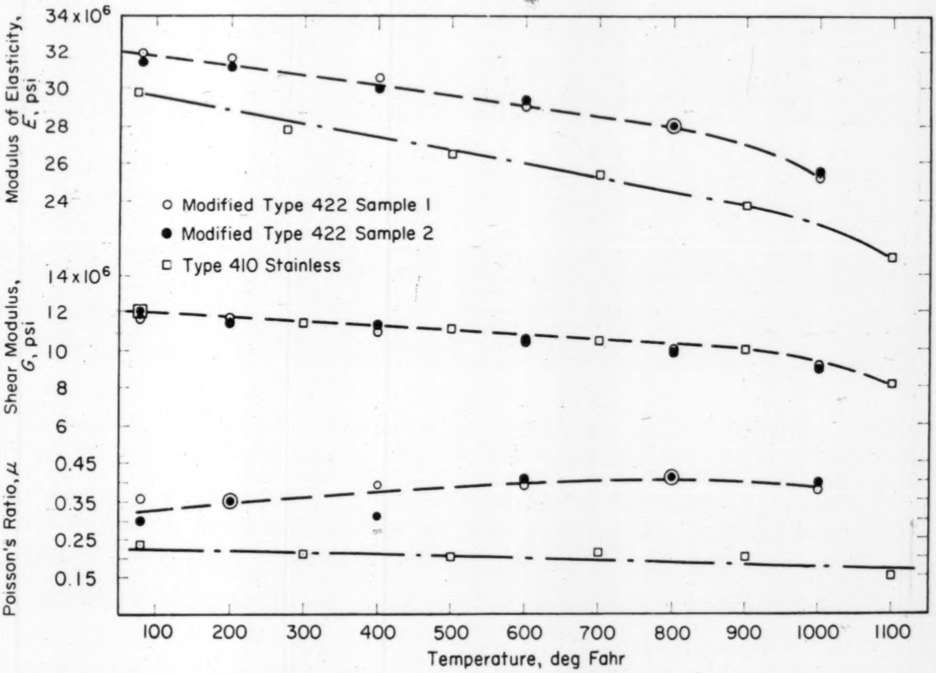


FIG. 4.—Variation with Temperature in the Elastic Moduli E, G, and μ for Modified Type 422 Stainless Steel and Type 410 Stainless Steel.

The test results for the modified type 422 steel are given in Table IV and shown graphically in Fig. 4, where the present results are compared with those for a type 410 steel. The values of Young's modulus for the modified type 422 steel are measurably greater than those of the type 410 steel for the entire temperature range covered. No observable difference is found in the shear modulus. As should be expected from the difference in Young's modulus, the computed values of Poisson's ratio for the modified type 422 steel are greater than those for the type 410 steel. It should be noted that the differences in the elastic moduli found between the modified type 422 steel and the type 410 steel are similar to those found between the Cr-Mn-V-N steel and the type 316 steel.

DISCUSSION OF RESULTS

The results obtained for the Cr-Mn-N steel, as indicated above, differ from the results obtained on the standard 300 series austenitic stainless steels. This is shown by the comparison with type 304 stainless steel which behaves in a manner similar to that of the other standard grades of stainless steel. The major differences in composition between the Cr-Mn-N steel and type 304 steel are in the amounts of manganese, nickel, and nitrogen each contains. Although the differences in manganese and nickel could conceivably affect the elastic properties of austenitic stainless steel, it is believed that the effects found in this study are due to dislocation pinning, primarily by nitrogen atoms. As may be observed from Table I, the Cr-Mn-N steel contains a large amount of nitrogen as well as a somewhat greater amount of carbon than type 304 steel. The lack of precipitation in the Cr-Mn-N steel, as shown from the microstructure in Fig. 1, indicates that these interstitial elements have been retained in solution.

The measured elastic moduli, *E* or *G*, depend on the ratio of the stress to the total strain, which is the sum of the lattice strain and the strain contributed by bulging of dislocation loops (6–10). Therefore Young's modulus, *E*, under a tensile load as indicated in the Appendix may be defined by

$$E = \frac{\sigma}{\epsilon_L + \epsilon_D} \qquad \text{(2)}$$

where σ is the applied tensile stress, ϵ_L the longitudinal lattice strain and ϵ_D the longitudinal strain contributed by bulging dislocation loops. Similarly the shear modulus, *G*, may be defined as

$$G = \frac{\tau}{\gamma_L + \gamma_D} \qquad \text{(3)}$$

where τ is the shear stress, γ_L the lattice shear strain and γ_D the shear strain contributed by bulging dislocation loops.

As shown in the Appendix the dependence of *E* and *G* on dislocation loop length, *l*, is given by

$$E = \frac{E_L}{1 + \left(\frac{1+\alpha}{1+\beta}\right)\frac{Nl^3}{6}}$$

$$G = \frac{G_L}{1 + \frac{Nl^3}{6}} \qquad \text{(4)}$$

where E_L and G_L are the lattice moduli in the absence of bulging dislocation loops, β and α are values of Poisson's ratio for the respective cases of strain caused by bulging dislocation loops and true lattice strain, and *N* is the number of dislocation loops of length, *l*, per unit volume. Equation 4 shows that both Young's modulus and the shear modulus increase as *l* decreases. Because of the interaction between interstitial atoms and edge dislocations it is likely that nitrogen and carbon atoms decrease the loop length by a pinning effect and cause an increase in the elastic moduli. Such an effect is believed to bring about the increase in *E* and the much smaller increase in *G* found for the Cr-Mn-N steel when compared to the results obtained for the type 304 steel. As may be observed from Fig. 2, the increase in E is appreciably greater than the increase in G. As a matter of fact, no difference is found in G for specimen No. 1. For this specimen, the increase in E is also smaller than for the second specimen. The greater change in E is also in agreement with a pinning effect causing a shortening in the dislocation loop lengths. From Eq 15 in the Appendix it is seen that following a reasonable approximation the ratio between the change in E and G is $\frac{E}{G}$. Since $E \approx 3G$, the change in E should be about three times the change in G. Thus the change in E should be more readily observable in the comparison made here between two different steels.

Of additional interest is the change in Poisson's ratio which depends on the change in both E and G. From Eq 19 in the Appendix it is observed that Poisson's ratio should not change with loop length. Although Eq 19 required an approximation, it can be shown more rigorously that the change in Poisson's ratio with loop length is at best very small. Thus, as may be observed from Fig. 2, this condition is satisfied by the results presented here. Except near room temperature the values of Poisson's ratio for both Cr-Mn-N and type 304 steels show little difference.

The fact that E and to some extent G for the Cr-Mn-N steel approach the values for type 304 steel as the temperature is increased, may also be explained qualitatively. As the temperature is increased, the interstitial atoms diffuse through the lattice more readily. Therefore, their pinning effect is diminished since they can move along with the dislocation loop as it is bulged. In effect, the loop length may become increasingly greater as the temperature is raised. It would be expected that at a sufficiently high temperature and beyond, the pinning effect should be essentially nil.

Depending on the interaction energy between the interstitial atoms and the dislocations, the effect on the elastic moduli may also be stress dependent. At a sufficiently high stress, even at low temperatures, the dislocation loops may be sufficiently bulged to become essentially unpinned, resulting in a lowering of the measured moduli under high stresses. The results presented in this study represent the behavior at low stresses not exceeding about 4000 psi.

As mentioned previously and as shown in Fig. 3, the results obtained for the Cr-Mn-Mo-V-N steel in the condition tested differ considerably from those obtained for the Cr-Mn-N steel. The behavior found for this steel, that is, the similarity between G values and the difference in E and μ as compared to type 316 steel, indicates that dislocation pinning is not a factor. This is consistent with the fact that the material was aged at 1300 F, thus promoting precipitation, as shown in Fig. 1, of nitrides and carbides so that carbon and nitrogen are not available in solid solution for dislocation pinning.

The only other possible explanation for the observed behavior of this steel is that the substitutional alloying elements present may, in some manner, affect E and μ. The elastic constants of metals and alloys depend on the cohesive forces between the atoms (**11**). The nature of the cohesive energy in metals, and particularly in alloys, is not well understood. In very general terms, the cohesive energy depends on the interaction between ions, on the energy and charge distribution of valency electrons, and on the Fermi electron energy. The effect of foreign atoms on these interactions is not known; however, it is suspected that the charge distribution and the Fermi energy level may be affected significantly by such atoms. The results obtained on the Cr-Mn-Mo-V-N steel indicate that this material is less compressible than the standard austenitic stainless steels. This is shown from the change in volume, ΔV, as follows:

$$\Delta V = 2\mu \epsilon - \epsilon \qquad (5)$$

where ϵ is the tensile strain. As μ approaches one half, the change in volume approaches zero. Thus, for this material the lattice contraction in the transverse direction under a tensile stress is increased, indicating some change in the electron charge distribution. Apparently such a change does not affect the overall cohesive energy in the case where dilatation effects are lacking, that is, under shear stresses only.

Of the two substitutional elements, manganese and vanadium, which are present in the Cr-Mn-Mo-V-N steel but not present to the same extent in standard austenitic stainless steels, vanadium may bring about the effect observed. Since the Cr-Mn-N steel did not behave in a similar manner, it would seem that manganese is not primarily responsible.

Additional evidence that vanadium may increase Young's modulus and Poisson's ratio is given by the results obtained on the modified type 422 steel. It is generally found in carbon and low alloy steels that Young's modulus, the shear modulus, and Poisson's ratio are unaffected by small additions of such elements as chromium or molybdenum (1). However, in modified type 422 steel an effect similar to that observed in the Cr-Mn-Mo-V-N steel has been found. In comparing the elastic moduli of modified type 422 stainless steel with those of a type 410 stainless steel it is seen from Fig. 4 that E and μ are appreciably greater for the modified type 422 stainless steel. It is unlikely that this effect could be attributed to molybdenum, since this element in carbon and low-alloy steels does not seem to affect the elastic moduli. It is likely then that in this case the effect may again be due to the presence of vanadium. This is substantiated by the fact that the behavior shown in Fig. 4 is identical to the behavior found in Fig. 3, where the results for the Cr-Mn-Mo-V-N steel are compared to those for type 316 austenitic stainless steel. Again it should be mentioned that it is not known whether these effects are stress dependent. The observations in this study apply only to relatively low stresses.

The foregoing discussion indicates that certain interstitial and substitutional elements in ferrite and austenite have a measurable effect on the elastic properties. In order to determine more clearly the nature of these effects, more detailed studies are needed.

SUMMARY

The temperature dependence of the elastic moduli of two austenitic stainless steels, a Cr-Mn-N steel and a Cr-Mn-Mo-V-N steel, and a modified type 422 ferritic stainless steel has been determined between room temperature and 1000 F. The elastic moduli and their temperature dependence of the austenitic steels differ in some respects from those of the usual chromium-nickel steels of the AISI 300 series. Young's modulus for the Cr-Mn-N steel is higher than that for a type 304 steel, although the difference decreases as the temperature is increased. The shear modulus is the same for one specimen and slightly greater for a second specimen. Except in the vicinity of room temperature Poisson's ratio is unchanged. The higher values of E and G found for the Cr-Mn-N steel are attributed to pinning of dislocation loops by nitrogen and carbon atoms. A qualitative, analytical substantiation of such an effect is offered. This analysis indicates that little change in Poisson's ratio should be found and agrees with the experimental observation.

In comparing the results obtained for the Cr-Mn-Mo-V-N austenitic stainless steel and for the modified type 422 ferritic stainless steel to the values for AISI types 316 and 410 steels, respectively, it is found that E is greater, G is unchanged but Poisson's ratio is also greater. The difference in both E and Poisson's ratio increases somewhat with increasing temperature. The similar effect found in both of these steels may be caused by a change in the valency electron charge distribution in the lattice brought about by the addition of vanadium.

Acknowledgment:

The help of R. R. Keck in conducting the moduli determinations is gratefully acknowledged. The help of O. Richmond and Y. T. Chou is also acknowledged. The specimens in the heat-treated and machined condition were supplied by the Applied Research Laboratory of U. S. Steel Corp.

APPENDIX

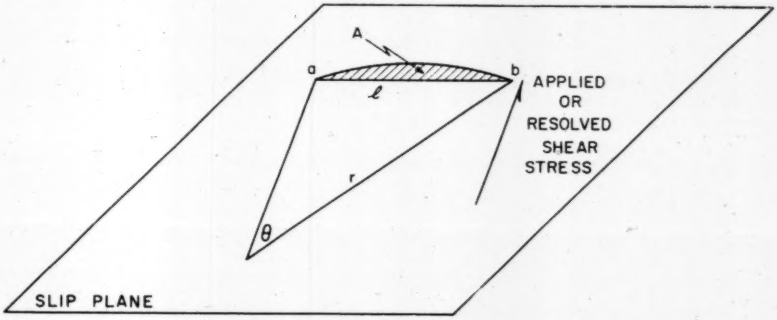


FIG. 5.—Schematic Drawing Showing Bulged Dislocation Loop.

The contribution to the measured strain by bulged dislocation loops may be determined as follows (**10**):

Assuming that the ends of a dislocation loop of length, l, are fixed in the slip plane at a and b, as shown in Fig. 5, the force per unit length for the bulged loop under a shear stress, τ, is $b\tau$, where b is Burger's vector. If the loop is bulged into a circular section of radius, r, the force per unit length becomes

$$b\tau = \frac{T}{r} \quad \ldots\ldots\ldots\ldots\ldots (6)$$

where T is the line tension. The area swept out by the bulged dislocation is $\frac{1}{2}\, r^2\ (\theta - \sin\theta)$, where r is the radius of curvature. For small values of θ the area, A, becomes

$$A = \frac{l^3}{12r} = \frac{l^3 b\tau}{12T} \quad \ldots\ldots\ldots (7)$$

If each loop traverses the same area for N loops of length, l, the shear strain becomes

$$\gamma_D = NAb = \frac{\tau N l^3}{6G_L} \quad \ldots\ldots\ldots\ldots (8)$$

where G_L is the lattice shear modulus.

The effect of loop length, l, on the measured shear modulus, G, may be determined as follows: The shear modulus is defined as the ratio of the stress to the total strain, which is the sum of the lattice strain and the strain contributed by bulging dislocation loops (**6–8**). Therefore,

$$G = \frac{\tau}{\gamma_L + \gamma_D} \quad \ldots\ldots\ldots\ldots (9)$$

where γ_L is the true lattice shear strain. By substituting Eq 8 in Eq 9 one finds:

$$G = \frac{G_L}{1 + Nl^3} \qquad (10)$$

A similar effect is found for Young's modulus. It can readily be shown that the maximum shear strain, γ, is related to the tensile strain, ϵ, by the relation

$$\epsilon = \frac{\gamma}{1 + \mu} \qquad (11)$$

where μ is Poisson's ratio. It follows therefore for small elastic strains that

$$\epsilon_L = \frac{\gamma_L}{1 + \alpha}$$

$$\epsilon_D = \frac{\gamma_D}{1 + \beta} \qquad (12)$$

where ϵ_L is the true lattice tensile strain and ϵ_D is the tensile strain resulting from bulged dislocation loops on planes of maximum resolved shear stress and α and β refer to Poisson's ratio for the two cases. The observed Young's modulus, E, may be defined as

$$E = \frac{\sigma}{\epsilon_L + \epsilon_D} \qquad (13)$$

where σ is the tensile stress. From Eq 8 and Eq 12, Eq 13 may be written

$$E = \frac{E_L}{1 + \left(\frac{1 + \alpha}{1 + \beta}\right)\frac{Nl^3}{6}} \qquad (14)$$

where E_L is the lattice Young's modulus.

The dependence of Poisson's ratio on dislocation loop length may be determined as follows: By differentiating Eq 10 and Eq 14 and combining one finds[4]

$$\frac{dE/dl}{dG/dl} = \frac{E}{G} \qquad (15)$$

The relation between μ, E and G may be written

$$2(1 + \mu) = \frac{E}{G} \qquad (16)$$

By differentiating Eq 16 with respect to l, one finds

$$2\frac{d\mu}{dl} = \frac{1}{G}\frac{dE}{dl} - \frac{E}{G^2}\frac{dG}{dl} \qquad (17)$$

and by combining Eq 17 and Eq 15 it is found that

$$\frac{d\mu/dl}{dG/dl} = 0 \qquad (18)$$

Therefore,

$$\frac{d\mu}{dl} = 0 \qquad (19)$$

Equation 19 has not been derived rigorously because of the assumption that $\left(\frac{1+\alpha}{1+\beta}\right) =$ 1. However, a more rigorous and lengthy derivation has shown that the change in μ with loop length is very small.

---

[4] In this operation it is assumed for simplicity that $\left(\frac{1 + \alpha}{1 + \beta}\right) = 1$. The value of this quantity is about 0.9.

REFERENCES

(1) T. S. Ke, "Stress Relaxation across Grain Boundaries in Metals," *Physiological Reviews*, Vol. 71, p. 533 (1947).

(2) C. D. Starr, E. C. Vicars, A. Goldberg, and J. E. Dorn, "Effect of Alloying Elements on Grain Boundary Relaxation in Alpha Solid Solutions of Aluminum," *Transactions*, Am. Soc. for Metals, Vol. 45, p. 275 (1953).

(3) F. Garofalo, P. R. Malenock, and G. V. Smith, "The Influence of Temperature on the Elastic Constants of Some Commercial Steels," Symposium on Determination of Elastic Constants, Am. Soc. Testing Mats., p. 10 (1952). (Issued as separate publication *ASTM STP No. 129*.)

(4) J. E. Dorn and T. E. Tietz, "The Modulus of Elasticity—A Review of Metallurgical Factors," *Metal Progress*, Vol. 81, July 1950.

(5) W. H. Graft, D. W. Levinson, and W. Rostoker, "The Influence of Alloying on the Elastic Modulus of Titanium Alloys," *Transactions*, Am. Soc. for Metals, Vol. 49, p. 263 (1957).

(6) F. Garofalo, "Survey of Various Tests Used to Determine Elastic, Plastic and Rupture Properties of Metals at Elevated Temperatures," *Journal of Basic Engineering*, Am. Soc. Mechanical Engrs., Paper No. 59-A-112, (1960).

(7) J. D. Eshelby, "Dislocations as a Cause of Mechanical Damping in Metals," *Proceedings*, Royal Soc (London), Vol. A197, p. 396 (1949).

(8) N. F. Mott, "A Theory of Work-Hardening of Metal Crystals," *Philosophical Magazine* (7), Vol. 43, p. 1151 (1952).

(9) J. Friedel, "Anomaly in the Rigidity Modulus of Copper Alloys for Small Concentrations," *Philosophical Magazine* (7), Vol. 44, p. 444 (1953).

(10) G. Schoeck, "Dislocation Theory of Plasticity of Metals," *Advances in Applied Mechanics*, Vol. IV, p. 229 (1956).

(11) M. A. Jaswon, "The Theory of Cohesion," Pergamon Press, Ltd., London (England) (1954).

DELAYED FAILURE OF HIGH-STRENGTH STEEL IN LIQUID ENVIRONMENTS

By E. A. Steigerwald[1]

Synopsis

The influence of liquid environments on the delayed failure characteristics of several high-strength steels was investigated. Aqueous solutions produced delayed failure in centrally precracked specimens of 300M and H-11 tool steel over an appreciable range of applied stresses. The failure process under constant load consisted of slow crack growth which ultimately produced total failure. The relationship between the crack size immediately preceding rapid propagation and the applied load agreed with current fracture mechanics concepts. The results indicate that the delayed failure in the presence of a liquid environment can occur with sufficient speed to produce a lowering of the notch tensile strength obtained in relatively slow strain rate tension tests.

The current quest for materials with higher usable strength has stimulated a considerable amount of work directed toward evaluation of material behavior in the presence of very sharp notches. The current fracture mechanics concepts (**1**,**2**)[2] provide a quantitative method for obtaining a basic material parameter (fracture toughness) which can be related to specimen and defect size in order to predict failure stresses. Due to the nature of the fracture analysis, the emphasis is placed on the relationship between the crack size and the applied stress at the instant of catastrophic failure. Relatively little information is available on the factors which govern the slow growth of a crack prior to rapid propagation.

The time-dependent delayed failure[3] of rocket motor cases at constant pressure when exposed to aqueous environments (**3**), the static fatigue of hydrogenated high-strength steel (**4**), and the delayed stress-corrosion cracking of aircraft steels (**5**) all indicate that the slow crack growth period is extremely sensitive to test conditions and may bear an important relationship to the failure of the total structure.

The object of this investigation was to evaluate systematically the influence of an aqueous environment on the delayed failure characteristics of high-strength steel. The influence of aqueous solutions on the slow crack growth process may also be important in affecting the parameters used to evaluate high-strength steels. The recommended method for fracture testing high-strength sheet materials involves ink staining techniques to determine the critical crack length (2). The question arises as to whether or not the stimulation of slow crack growth by the staining media has any influence on the fracture toughness parameter or on the notch tensile strength.

---

[1] Research Metallurgist, Materials Research and Development, Thompson Ramo Wooldridge, Inc., Cleveland, Ohio.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

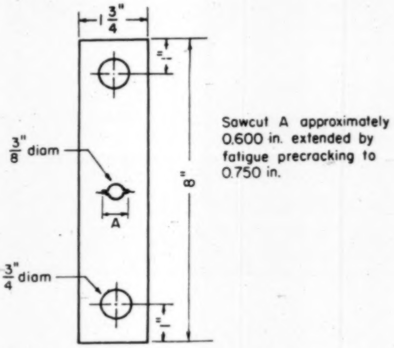
[3] Delayed failure is defined as the property of a material to fail under the action of a sustained load even though it is capable of supporting a higher load for a finite period of time.

MATERIALS AND PROCEDURE

A low alloy martensitic steel designated as 300M and a H-11 type hot-work tool steel were used in the investigation. The compositions of these steels are presented in Table I. The 300M material was used in the as-received thickness while the H-11 steel was ground to 0.085 in. by removing equal amounts of material from both surfaces prior to heat treatment in order to remove a heavy surface scale.

TABLE I.—COMPOSITION OF TEST MATERIAL.

Material	Chemical Composition, per cent by weight							Sheet Thickness (As-Received), in.
	Carbon	Manganese	Silicon	Chromium	Nickel	Vanadium	Molybdenum	
300M	0.43	0.89	1.78	0.90	1.92	0.13	0.43	0.080
H-11	0.39	0.30	1.00	5.14	...	0.49	1.30	0.097



Sawcut A approximately 0.600 in. extended by fatigue precracking to 0.750 in.

FIG. 1.—Center Precracked Notch Tension Specimen.

The delayed failure properties of the material were determined with centrally-notched and precracked tensile specimens (Fig. 1). The specimen preparation was similar to that used by Srawley and Beachem (6). A blank $1\frac{3}{4}$ in. wide was sheared from the sheet stock and a center notch 0.600 in. in length was produced with a jeweler's saw. Subsequent to heat treatment the specimens were precracked in tension-tension fatigue to produce a $\frac{3}{4}$-in. center notch. The exact precrack dimension was measured with a toolmaker's microscope after failure. The 300M was hardened by austenitizing at 1700 F in salt and air cooling, followed by heating to 1600 F in salt and oil quenching. Tempering temperatures of 600, 1025, and 1150 F were employed with double 1-hr cycles. The H-11 tool steel was austenitized at 1850 F in salt, air cooled, and double tempered for 2-hr cycles at 1000 F. The conventional tensile properties produced by these heat treatments are presented in Table II.

TABLE II.—TENSILE PROPERTIES OF 300M AND H-11 STEELS USED.

Material	Tempering Temperature, deg Fahr	0.2 per cent Yield Strength, psi	Tensile Strength, psi	Elongation in 2 in., per cent
300M	600	245 000	295 000	6.0
300M	1025	205 000	226 000	8.5
300M	1150	168 000	187 000	11.0
H-11	1000	235 000	290 000	5.0

Constant load, stress-rupture tests were conducted on the centrally pre-

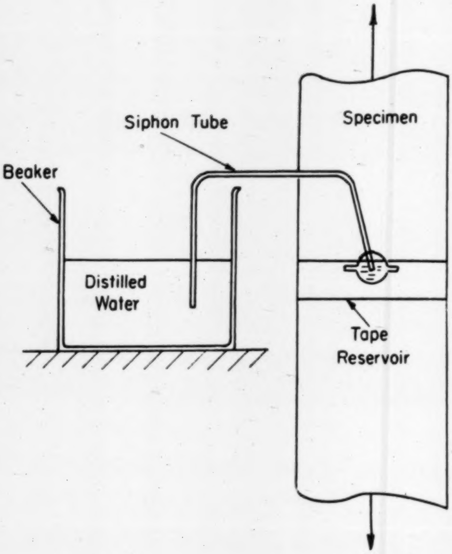


FIG. 2.—Schematic Representation of Experimental Setup.

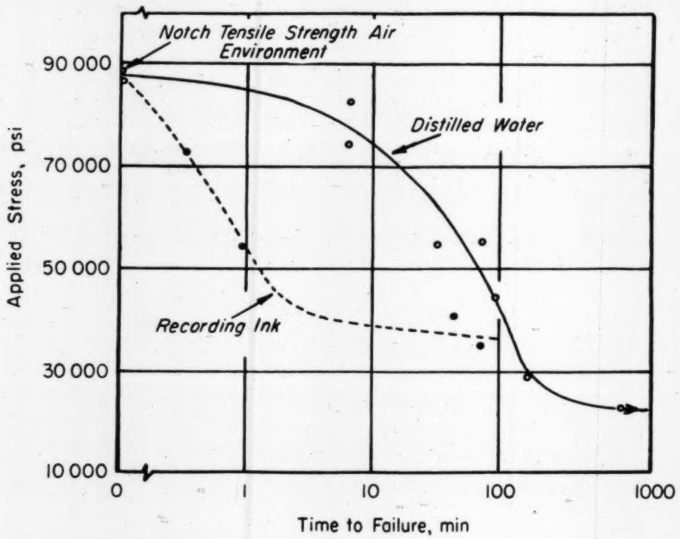


FIG. 3.—Delayed Failure of 300M Steel Center Precracked Specimens at 295,000-psi Strength Level.

cracked specimens. Figure 2 presents a schematic diagram of the test setup. The crack was surrounded by liquid environments with the aid of a tape reservoir and the reservoir was maintained at a constant level during long-time tests by a beaker and siphon arrangement. Short-time tests were also conducted without the use of tape in an effort to determine if any significant contamination effects were produced by the tape container. The failure times were not altered by this procedure. In the testing sequence the liquid was applied to the specimen immediately after application of the load. Resistance measurements using a previously described method (7) were employed to chart the crack growth kinetics for a particular set of specimen conditions.

As a standard of comparison, constant load tests were also conducted on precracked specimens in which no liquid was applied. Under this condition failure did not occur after a 100-hr test time at applied loads which represented 85 per cent of the conventional notch tensile strength.

RESULTS AND DISCUSSIONS

Delayed Failure in 300M and H-11:

Figure 3 illustrates the results of delayed failure tests on 300M tempered at 600 F (295,000 psi tensile strength). The curves are presented with applied stress, defined as the load divided by the initial cross-sectional area in the plane of the notch, indicated along the ordinate, and with the logarithm of the time to failure on the abscissa. The notch tensile strength plotted at zero time corresponds to the strength obtained in a conventional notch tension test conducted without a liquid media in the crack and at a crosshead movement of 0.05 in. per min. The results of Fig. 3 indicate that both recording ink and distilled water produced a considerable range of delayed failure. The most noteworthy aspect, however, is the relatively rapid rate at which the crack propagated in the presence of the liquid environment. At a nominal stress of 55,000 psi failure occurred in less than 1 min when recording ink was placed in the crack. The speed of the delayed failure indicates that the notch tensile strength parameter can be strain rate dependent, that is, decrease with decreasing strain rate. Under the proper conditions of environment and heat treatment the time for a conventional tension test can be sufficiently slow to allow the liquid media to have an effect.

Figure 4 shows the delayed failure curve for 300M steel tempered at 1025 F (226,000 psi strength level) and at 1150 F (187,000 psi strength level). In the case of the material tempered at 1025 F the range of applied stress over which delayed failure occurred was considerably greater than for the 600 F temper. The increase of the conventional notch tensile strength (zero time) produced by the 1025 F temper was the principal cause for the extended failure range. The time to failure for the 300M steel at this strength level was somewhat greater than that required for the 600 F temper.

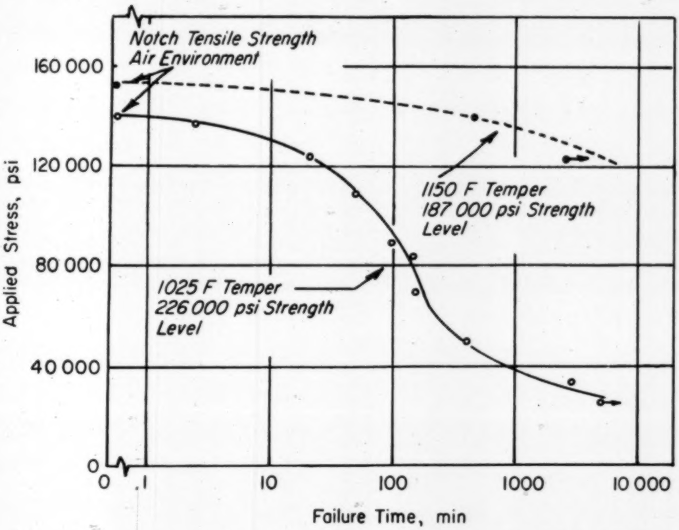


FIG. 4.—Delayed Failure of 300M Steel Center Precracked Specimens Subjected to a Distilled Water Environment.

The limited data on 300M tempered at 1150 F indicated that the material in this condition was extremely resistant to delayed failure and approximately 4 hr were required for failure at 140,000 psi.

It is interesting to note the qualitative correlation between the normal notch strength of the material and its tendency for delayed failure. As the material became more insensitive to notch effects

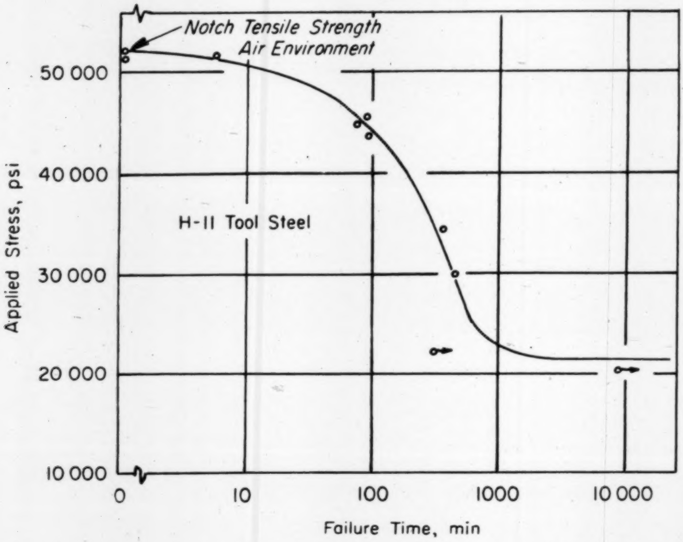


FIG. 5.—Delayed Failure of H-11 Tool Steel, Heat Treated to a 295,000-psi Strength Level, Center Precracked Specimens Subjected to a Distilled Water Envroniment.

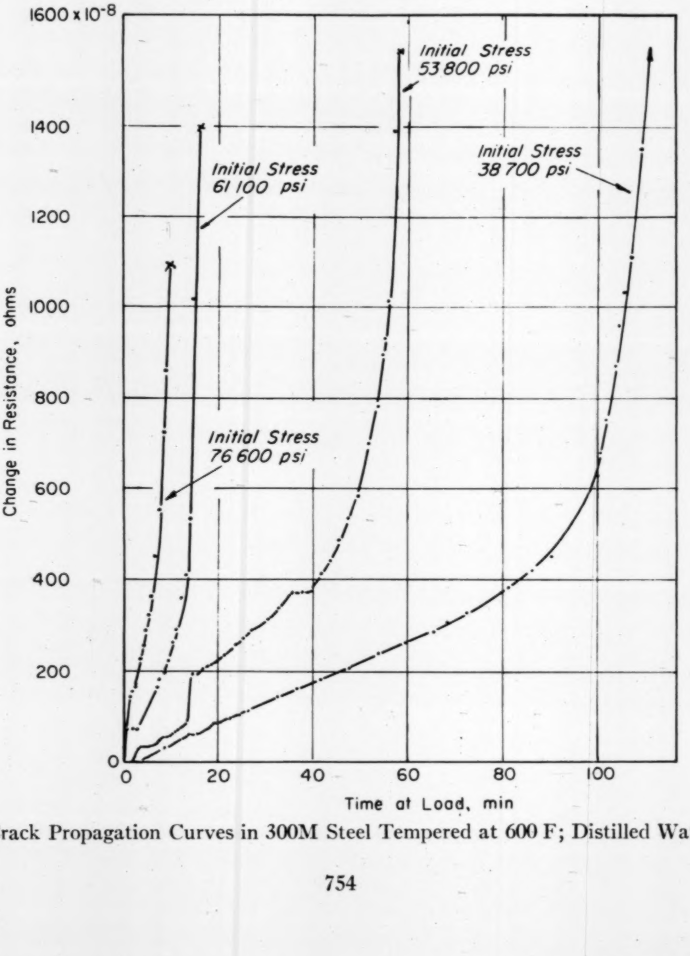
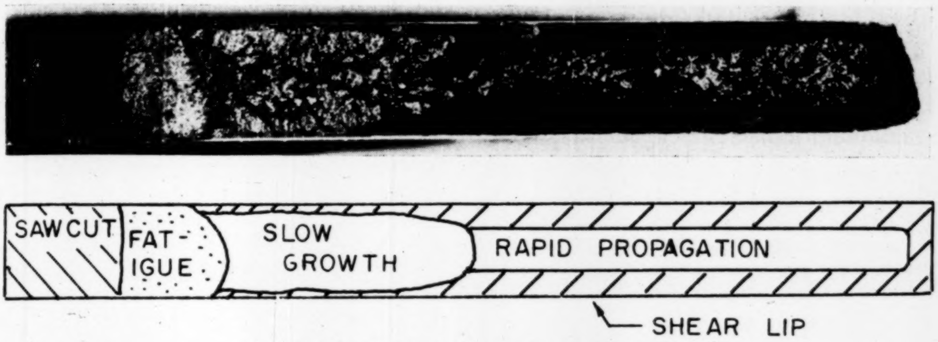
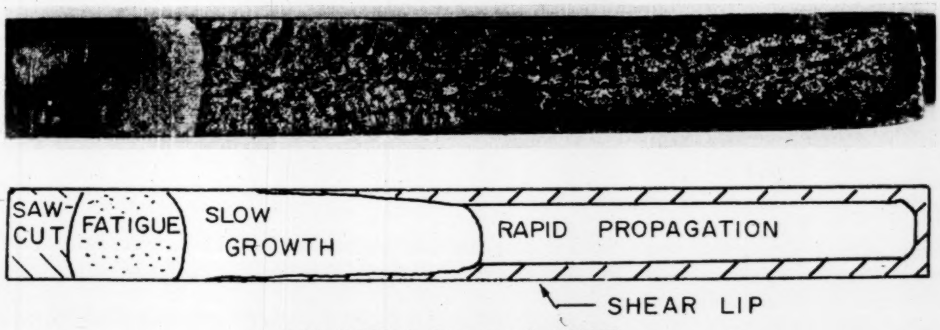


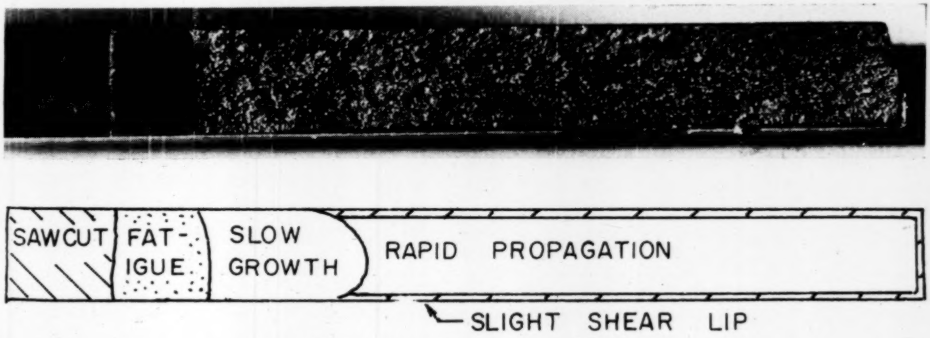
FIG. 6.—Crack Propagation Curves in 300M Steel Tempered at 600 F; Distilled Water Environment.


SAWCUT
FATIGUE
SLOW GROWTH
RAPID PROPAGATION
SHEAR LIP

(*a*) 300M steel tempered at 1025 F.


SAW-CUT
FATIGUE
SLOW GROWTH
RAPID PROPAGATION
SHEAR LIP

(*b*) 300M steel tempered at 600 F.


SAWCUT
FATIGUE
SLOW GROWTH
RAPID PROPAGATION
SLIGHT SHEAR LIP

(*c*) H-11 tool steel.

FIG. 7.—Fracture Appearance and Schematic Interpretation of Delayed Failure Specimens; Distilled Water Environment (×9).

the delayed failure tendency decreased. These results are analogous to those obtained for delayed failure in high-strength steels due to hydrogen (**4**) and to stress corrosion (**5**).

The delayed failure of H-11 steel in the presence of distilled water is presented in Fig. 5. The general nature of the behavior was similar to that observed for the 300M steel. Delayed failure tests with a distilled water crack environment were also conducted on

0.050 in. thick B120VCA titanium sheet. The specimen geometry and preparation were identical to those employed for steel. The titanium was received in the solution treated condition and aged for 24 hr at 900 F to produce a smooth tensile strength of 170,000 psi. The notch tensile strength was 95,000 psi. The water environment did not produce delayed failure in this material in a 100-hr test period at an applied stress of 80,000 psi.

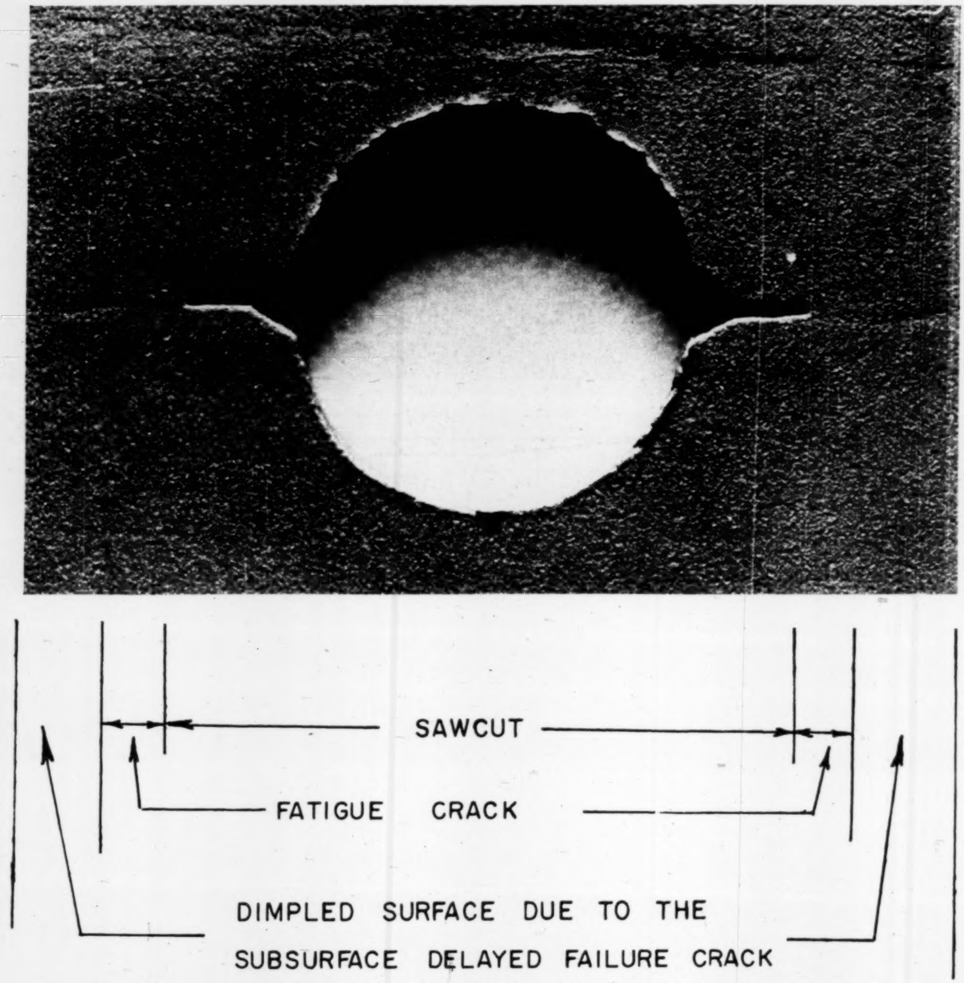


FIG. 8.—Macroscopic Appearance of Crack Growth During Failure Process in 300M Steel Tempered at 1025 F (×6).

Nature of the Slow Crack Growth:

The kinetics of the slow crack growth process were studied with resistivity measurements and Fig. 6 indicates the nature of the crack growth process for 300M steel tempered at 600 F. The initial stage of growth was featured by the apparent absence of a definite incubation time. It should be noted, however, that incubation times less than 1 min are relatively difficult to identify with the particular method employed. At the higher applied stresses the growth was relatively rapid while at the lower loads a very slow growth initially occurred; this was followed by the faster crack extension, ultimately leading to catastrophic failure. All the curves exhibited some discontinuities and these discontinuities were even more obvious through audible clicking and continuous observation in the latter stages of the growth period.

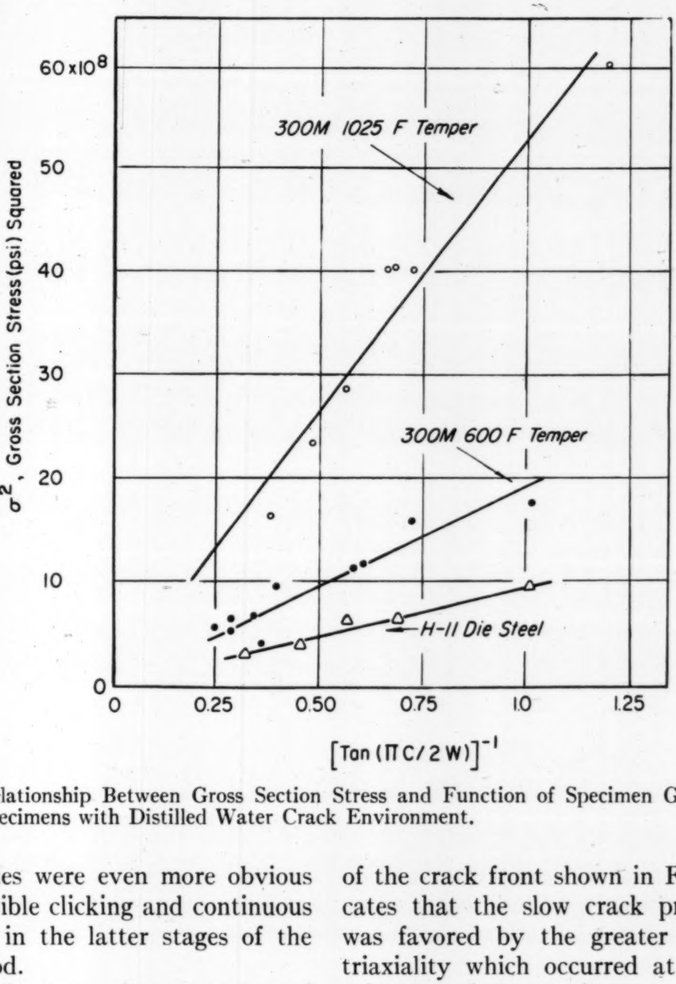


FIG. 9.—Relationship Between Gross Section Stress and Function of Specimen Geometry for Precracked Specimens with Distilled Water Crack Environment.

Figure 7 illustrates the appearance of both the slow crack growth promoted by the distilled water and the catastrophic growth which occurred when the crack had presumably attained a critical size. The difference between the two fracture modes was particularly obvious in the 300M steel where the normal crack propagation was accompanied by an appreciable shear lip. Figure 8 illustrates the external appearance of a crack in progress. The slow cracking became apparent by a "dimpling" of the section ahead of the fatigue crack. This result coupled with the parabolic appearance of the crack front shown in Fig. 7 indicates that the slow crack propagation was favored by the greater degree of triaxiality which occurred at the mid-thickness of the specimens.

Since the embrittling action of the environment would be expected to cause only a local instability of the crack in the immediate vicinity of the crack tip, the transition from slow to catastrophic crack growth should be governed by the same fracture toughness which is measured without the embrittling action of the environment. If this reasoning is correct the fracture toughness values obtained by ink staining would not be influenced by the staining media or the time at load since the lower applied load at failure would be compensated by the larger critical crack length. The relationship between stress and critical crack length can be expressed as (1,2):

$$K_c^2 = \sigma^2 W \tan\left(\frac{\pi C}{2W}\right) = \mathcal{G}_c E \quad \ldots\ldots (1)$$

where:

$\mathcal{G}_c$ = strain energy release rate for onset of catastrophic failure,
K_c = fracture toughness,
W = specimen width,
E = Young's modulus,
C = total crack length at moment of failure determined from fracture appearance, and
σ = gross stress on specimen at distance removed from the crack.

This expression neglects the plastic zone correction (2), hence would be expected to give values of fracture toughness below the true value for the particular material. Since the specimen width is essentially constant, a plot of σ^2 as a function of the reciprocal tan $(\pi C/2W)$ will be linear and extrapolate through the origin if K_c is a true constant and not influenced by the test time. Figure 9 represents the relationship between gross section stress and critical crack length for both the 300M and H-11 steels. The relationship is essentially linear and therefore agrees with that required by Eq 1. The results presented in Fig. 9 would be expected to deviate from linearity at high values of C (low values of the reciprocal function) since Eq 1 becomes less accurate as the parameter $C/2W$ increases (1). Both the 300M steel tempered at 600 F and the H-11 tool steel conformed to the linear function over the entire range investigated while the 300M tempered at 1025 F deviated slightly from the accepted relationship at the high values of crack length.

Since the fracture toughness, K_c, remains constant over the delayed failure range the ink staining techniques introduce no error in fracture toughness evaluations. Any decrease in applied stress produced by the staining environment is attended by an increase in critical crack length. Depending on test conditions, the staining techniques involving aqueous solutions can reduce the notch tensile strength; therefore a danger may exist in attempting to determine both K_c and the notch tensile strength (notch strength ratio) from a single specimen. Since the staining solutions do influence the stress necessary for the initiation of a crack, these techniques should be used with caution when applied to determine energy release rates for the conditions governing crack initiation and slow crack growth.

TABLE III.—INFLUENCE OF VARIOUS CRACK ENVIRONMENTS ON FAILURE TIME OF 300M STEEL.

Environment	Failure Time, min
Recording ink	0.5
Distilled water	6.5
Amyl alcohol	35.8
Butyl alcohol	28.0
Butyl acetate	18.0
Acetone	120
Lubricating oil	150
Carbon tetrachloride	no failure in 1280
Benzene	2247
Air	no failure in 6000

In an effort to obtain a staining media which would not produce delayed failure and to gain some insight into the mechanism which may be operative, the delayed failure characteristics of 300M steel tempered at 600 F were investigated in the presence of various liquid environments. Table III lists the liquids employed and the observed failure times.

The specimens in all cases were loaded to a notch tensile stress of approximately 75,000 psi which represented 83.5 per cent of the strength obtained in a conventional notch tension test without a liquid crack environment.

The pH of aqueous solutions was also varied from 4.8 to 9.0 with the use of buffered solutions; however, this variation produced no significant difference in the failure time of 300M steel.

Nonaqueous solvents consistently caused delayed failure; however, the times were considerably longer than those produced by the aqueous environments. Although no direct correlation was apparent between the nature of the environment and the failure time, the solvents with closed ring structures and low dielectric constants, benzene and carbon tetrachloride, were capable of sustaining the applied stress for the greatest time periods.

At present, the delayed failure induced by crack environment can be attributed to three possible factors: (1) lowering of the surface energy of the crack due to liquid adsorption, (2) hydrogen embrittlement, and (3) stress corrosion. The data presented are not sufficient to allow the particular mechanism or the combination of factors which are producing the embrittlement to be conclusively defined. The surface adsorption mechanism has been widely used to explain delayed failure phenomena in materials (8), and the apparent absence of an incubation time is a necessary, but not sufficient, condition for a surface adsorption mechanism. A test was conducted on a 300M specimen that was not precracked but merely had a center sawcut with a terminal radius of approximately 0.005 in. The specimen was loaded to 90 per cent of the normal notch tensile strength and subjected to a distilled water environment. Failure occurred in 15.2 hr. In this case it is difficult to visualize how simple surface adsorption of the liquid on the face of the sawcut could lower the failure stress. Since the sawcut itself does not extend and a crack must be initiated, another mechanism besides surface adsorption appears to be rate controlling.

Although hydrogen embrittlement and stress corrosion have been observed in the high-strength steels, the manner in which sufficient hydrogen is produced or stress corrosion takes place in the various environments requires description before these factors can be used to explain the observed delayed failure process.

SUMMARY

Notched precracked specimens of high-strength steel are extremely susceptible to delayed failure when exposed to liquid environments. The time in which failure occurs is a function of applied loads and, in the case of steels heat treated to high strength levels, the failure can occur in a matter of minutes at stresses as low as 75 per cent of the normal notch tensile strength. The tendency for delayed failure decreases as the strength level of the steel decreases. Although aqueous solutions are the most potent environments for producing the failure process, organic solvents will also cause the embrittlement.

The delayed failure occurs by a process of slow crack growth until the crack reaches a critical, unstable length where catastrophic failure occurs. The concepts of fracture mechanics adequately describe the relationship between applied stress and critical crack size. The results indicate that ink staining techniques employed to determine critical crack lengths will not alter values of fracture toughness. The delayed failure process, however, can occur with sufficient speed so that the notch tensile strength may be lowered by environment. This decrease in strength can be manifested, for example, in the time-dependent failure of rocket chambers during pressure testing. The selection of a suitable proof testing technique can therefore be an important consideration to proper component evaluation.

REFERENCES

(1) G. R. Irwin, J. A. Kies, and H. L. Smith, "Fracture Strengths Relative to Onset and Arrest of Crack Propagation," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 640 (1958).

(2) ASTM Committee Report, "Fracture Testing of High-Strength Sheet Materials," ASTM BULLETIN, No. 243, Jan., 1960, p. 29.

(3) M. E. Shank, C. E. Spaeth, V. W. Cooke, and J. E. Coyne, "Solid-Fuel Rocket Chambers for Operation at 240,000 psi and Above," *Metal Progress*, Vol. 76, No. 5, and Vol. 76, No. 6, Nov. and Dec., 1959.

(4) A. R. Troiano, "The Role of Hydrogen and Other Interstitials in the Mechanical Behavior of Metals," Campbell Memorial Lecture, *Transactions*, Am. Soc. Metals, Vol. 52, p. 54 (1960).

(5) E. H. Phelps and A. W. Loginow, "Stress Corrosion of Steels for Aircraft and Missiles," *Corrosion*, Vol. 16, No. 7, p. 97, July, 1960.

(6) J. E. Srawley and C. D. Beachem, "Crack Propagation Tests of Some High Strength Sheet Steels," NRL Report No. 5263, Jan. 10, 1959.

(7) W. J. Barnett and A. R. Troiano, "Crack Propagation in the Hydrogen Induced Brittle Fracture of Steel," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 209, p. 486 (1957).

(8) N. J. Petch and P. Stables, "Delayed Fracture of Metals Under Static Load," *Nature*, Vol. 169, p. 842, May 17, 1952.

EFFECT OF CARBON CONTENT AND MELTING PRACTICE ON ROOM-TEMPERATURE SHARP-EDGE-NOTCH TENSILE CHARACTERISTICS OF H-11 MODIFIED AND 300M SHEET STEELS*

By J. L. Shannon, Jr.,[1] G. B. Espey, [1]A. J. Repko,[1] and W. F. Brown, Jr.[2]

Synopsis

The effects of carbon content and the possible advantages of consumable electrode vacuum remelting on the room-temperature smooth and sharp-edge-notch tensile strengths and the fracture toughness (K_{c2}) of H-11 modified and 300M alloys were investigated. A wide range of tempering temperatures was studied for each alloy.

As expected, the smooth tensile strength of both alloys increased with carbon content. This strengthening effect decreased at the highest tempering temperatures. The influence of the carbon content on the notch sensitivity is distinctly different for the two alloys. 300M alloy exhibited fully notch ductile behavior, for the particular notch specimen used, at strength levels below 200,000 psi for all carbon levels investigated. At higher strength levels, notch sensitivity increased with increasing carbon. H-11 modified alloy exhibited fully notch ductile behavior below 160,000 psi for the range of carbon contents investigated. At strength levels between about 175,000 to 250,000 psi the notch sensitivity decreased with increasing carbon. The highest strength levels obtainable in this alloy with any carbon content have approximately equal and high notch sensitivity.

Consumable electrode vacuum remelting had no apparent effect on the smooth tensile strength of either alloy. Its effect on the notch sensitivity was only slight and faded out at the lower strength levels.

There is a pressing need to increase the fracture toughness of high-strength heat-treated ferritic sheet steels. Presently available commercial alloys develop notch sensitivity rapidly as tensile strength levels increase above 200,000 psi (**1**,**2**).[3] At tensile strength levels approaching 300,000 psi, fracture toughness values (**2**,**3**) are so low that extreme care must be used to avoid very small flaws that may initiate failure in highly stressed components. An appreciable increase in the fracture toughness would increase reliability, allow more flexibility in design, and permit more economical fabrication methods. It is suspected that low fracture toughness in these steels is in part associated with the following factors: 500 F embrittlement, the presence of certain minor elements, and the size and distribution of inclusions. In turn, these factors are influenced by both composition and melting practice. This paper is concerned with the effects of carbon variation and the possible advantages of consumable electrode vacuum remelting on the room-temperature notch sensitivity of a low-alloy ultra-high-strength steel and a tool steel.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Research Engineer, Strength of Materials Branch, National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio.

[2] Chief, Strength of Materials Branch, National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

Regarding the effect of carbon content, Schwartzbart and Sheehan (4,5) have reported Charpy V-notch transition temperature data for several low-alloy steels. At Rockwell hardnesses above about C 50, the transition temperature of SAE steel types such as 1300, 2300, 3100, 4100, 4300, 4600, 5100, and 8600 generally increased with increasing carbon content. However, the magnitude of the effect varied with the composition. At lower hardness levels no consistent effects of carbon content were established. Similar results were recently obtained by Klier (6) in notch tension testing of SAE 4300-V modified sheet steels having various carbon contents. A small amount of information (7,8) indicates that the room-temperature notch strength of cylindrical specimens may be improved by reducing the carbon content of low-alloy steels heat treated to high strength levels. In contrast, Hamaker and Vater (9) have recently shown that the room-temperature V-notch Charpy impact strength of H-11 modified steel decreases with decreasing carbon content when the steel is heat treated to tensile strength levels above 250,000 psi.

Vacuum melting is known to increase cleanliness (10) and reduce gas content. The possibility of a consequent improvement in mechanical properties has been the subject of numerous investigations. There is no doubt that vacuum remelting, particularly by the consumable electrode process, improves the properties of super alloys (10,11). Furthermore, there appears to be ample evidence that the fatigue strength of low-alloy and bearing steels is increased by vacuum melting (12,13,14). However, there is conflicting evidence regarding the influence of vacuum melting on the toughness of heat treated steels as measured by impact tests or notch tension tests. According to Cvikevich (15), only small improvement in the Charpy impact values is obtained by consumable electrode vacuum remelting of aircraft quality SAE 4340 steel. These small effects were confined to conditions above 700 F temper. Zima (16) reported essentially no improvement in the low-temperature impact properties of 300M type steel due to either induction or consumable electrode vacuum melting. On the other hand, an investigation by Gensamer (17) indicates that the 550 F embrittlement, as revealed by −320 F tensile reduction in area, can be eliminated in SAE 1340 steels by vacuum melting using a special molecular still principle. Blanchard and Troiano (18) indicate that both the notch tensile and delayed failure properties of high-strength SAE 4340 are improved by induction vacuum melting. On the other hand, Raring and Rinebolt (19) report that the susceptibility of SAE 4340 steel to delayed failure using sharply notched specimens is unaffected by induction vacuum melting. One very recent investigation by Bernstein and Young (20) provides fracture toughness data for high-strength sheet steels both air and consumable electrode vacuum melted. The results show negligible improvement produced in 300M by vacuum melting but indicate substantial increases in fracture toughness for AMS 6434.

Only two of the previously discussed investigations provide information directly related to the sharp notch sensitivity of high-strength sheet alloys. As mentioned, much of the evidence regard-

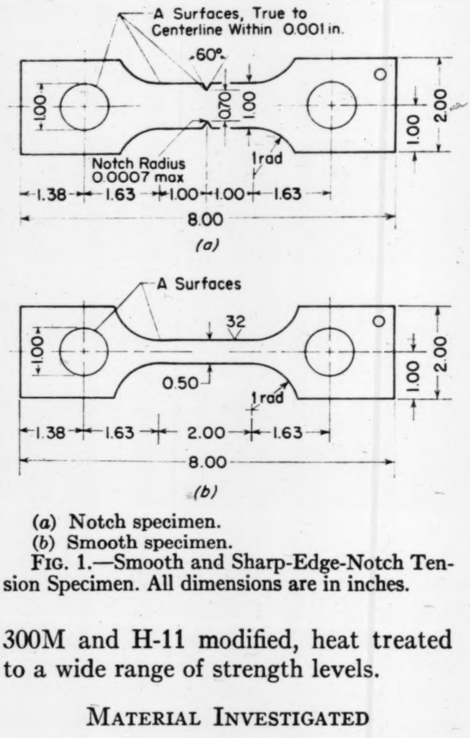
TABLE I.—CHEMICAL COMPOSITION OF SHEET ALLOYS INVESTIGATED.

Alloy	Supplier	Melting Practice	Heat Number	Heat Size, lb	Ingot Size, in.	Composition, per cent									
						Melt Carbon	Sheet Carbon	Silicon	Manganese	Sulfur	Phosphorus	Chromium	Vanadium	Molybdenum	Nickel
H-11 modified	Vanadium Alloys	Air-melt	31296	28 000	10 to 15 (square)	0.24	0.23	0.89	0.32	0.017	0.016	4.91	0.54	1.30	...
		Air-melt	31315	28 000	10 to 15 (square)	0.34	0.26	0.97	0.29	0.010	0.012	5.14	0.45	1.28	...
		Air-melt	B1290	300	7 (square)	0.30	0.29	0.92	0.35	0.026	0.012	5.03	0.58	1.21	...
		CEVM[a]	05059	7 504	24 (round)	0.39	0.34	0.84	0.26	0.010	0.010	4.85	0.48	1.28	...
		Air-melt	31682	25 000	15 (square)	0.39	0.39	0.91	0.33	0.010	0.017	5.02	0.52	1.22	...
		Air-melt	30625	28 000	10 to 15 (square)	0.43	0.43	0.93	0.31	0.011	0.017	5.24	0.55	1.28	...
300M	International Nickel	Air-melt	25996[b]	100	4 (square)	0.33	0.28	1.58	0.54	0.003	0.025	0.82	0.08	0.40	2.0
		Air-melt	25997[b]	100	4 (square)	0.38	0.34	1.65	0.64	0.009	0.023	0.84	0.08	0.41	2.0
		Air-melt	25998[b]	100	4 (square)	0.44	0.40	1.62	0.69	0.005	0.025	0.83	0.08	0.43	2.0
	Allegheny-Ludlum	CEVM	22320	1 300	12 (round)	0.44	0.45	1.73	0.65	0.009	0.011	0.92	0.10	0.32	1.85
		Air-melt	40775	5 000	12 (round)	0.44	0.46	1.70	0.81	0.009	0.010	0.93	0.14	0.32	1.88

[a] Consumable electrode vacuum remelt.
[b] Rolled by Armour Research Foundation.

ing melting practice is conflicting. This is not surprising since it is difficult to avoid differences in chemical composition that may mask the effects characteristic only of the melting practice.

The present investigation is an attempt to establish the influence of carbon content and to explore further the effect of consumable electrode vacuum remelting on the room-temperature notch sensitivity of two popular missile casing steels, 300M and H-11 modified, heat treated to a wide range of strength levels.



(*a*) Notch specimen.
(*b*) Smooth specimen.
FIG. 1.—Smooth and Sharp-Edge-Notch Tension Specimen. All dimensions are in inches.

MATERIAL INVESTIGATED

The composition, melting practice, heat size, and ingot size of the aircraft quality alloys investigated are listed in Table I. All alloys were cross rolled from the sheet bar to a nominal thickness of 0.063 in. except the three heats of 300M alloy supplied by the International Nickel Co. The latter were hand rolled to 0.80 in., roller leveled, and then surface ground to 0.063 in. Attempts were made by the alloy producers to control the carbon content within specified limits and to produce minimum variation in other elements from heat to heat of each alloy type. This is difficult to accomplish, particularly when close composition matching is attempted between air and consumable electrode vacuum remelted material. It was desired to investigate the influence of melting practice on 300M at both a high and intermediate (0.35 per cent) carbon level. Unfortunately, consumable electrode vacuum remelted 300M specified at 0.35 per cent carbon showed severe surface decarburization. The resulting sheet carbon content was only 0.22 per cent. This heat was therefore excluded from the investigation.

Carbon analyses[4] were made on fully heat-treated specimens of each composition. The results reported in Table I represent the average of at least six specimens selected from different positions in the sheet. In reporting the results, use is made of sheet carbon values rather than the melt carbon content as is usually done. According to Table I some heats exhibited significantly lower sheet carbon than melt carbon. A hardness study described in the Appendix revealed that certain 300M heats had a mild carbon gradient through the sheet thickness. As discussed in the Appendix, this gradient apparently did not significantly influence the results.

Inclusion ratings were not available for all of the heats. However, where information was furnished, it indicated high cleanliness in both air and vacuum-melted materials, typical of aircraft quality stock. As discussed previously (1), the authors feel that it is exceedingly difficult to correlate the fracture behavior with inclusion ratings, particularly when the steels in question are very clean.

---

[4] Carbon analyses were by the gravimetric method with an accuracy of approximately ±3 per cent. Chips for analysis were sheared from the specimens in order to avoid the possible loss of carbon caused by burning of chips during drilling or milling.

PROCEDURE

Details regarding the design and preparation of sharp-edge-notch tension specimens for screening purposes are described in the recent report of the ASTM Committee on the Fracture Testing of High-Strength Sheet Materials (2). The notch specimen employed in this investigation (Fig. 1) is basically that described by the ASTM committee, with the exception that notch radii were held to 0.0007 in. maximum instead of the 0.001 in. mentioned in the committee report. The smooth specimen used is also shown in Fig. 1. For both specimens, machining was confined to the sheet edges, since pressure vessels are not normally subjected to any surface machining. An exception was the 0.080-in. sheet of 300M alloy. This was reduced to 0.063 in. by surface grinding in order to remove severe surface roughness and to eliminate a possible thickness effect (2).

TABLE II.—HEAT TREATMENT SCHEDULE FOR SHEET ALLOYS INVESTIGATED.[a]

Alloy	Heat Treatment
H-11 modified	1850 F, ½ hr; air: triple temper 3-hr total Temper range: 900 to 1200 F
300M	1700 F, ½ hr; furnace-cool to 1600 F, ½ hr; oil: 1-hr temper Temper range: 400 to 1150 F (except for 0.46 per cent C 300M, which was tempered 400 to 1300 F)

[a] All high-temperature treatments in argon and all tempering in air.

Heat treatment followed finish machining and degreasing according to the schedule shown in Table II. Austenitizing was done in argon and tempering in air. Details of the machining (2,22) and heat treating (1) procedure have been described previously. Hardness values were determined on selected specimens after austenitizing and on every specimen after tempering.

Sharp-edge-notch and smooth tension tests were made for each composition and heat treatment. In all cases, the range of tempering temperatures investigated produced the strongest (brittlest) conditions as well as strength levels yielding fully notch ductile behavior (notch strength equal to or above the tensile strength). The notch strength at each tempering temperature was established by at least duplicate, and in most cases, triplicate tests. Both the longitudinal and transverse directions were tested on several heats of each alloy type, and no significant directionality effects were observed (see Figs. 2 (*a*) and (*b*)). Inasmuch as insufficient material was available to permit tests in both directions on all heats, only longitudinal properties are compared in this paper.

Details of the room-temperature testing procedure have been described previously (2). In all cases, attempts were made to determine the crack lengths at the onset of fast fracture in notch specimens by the use of ink staining as suggested by Irwin and his coworkers (2). The analysis of these data to determine fracture toughness (K_{c2}) values follows the recommendations of the ASTM Committee on Fracture Testing of High-Strength Sheet Materials (2).

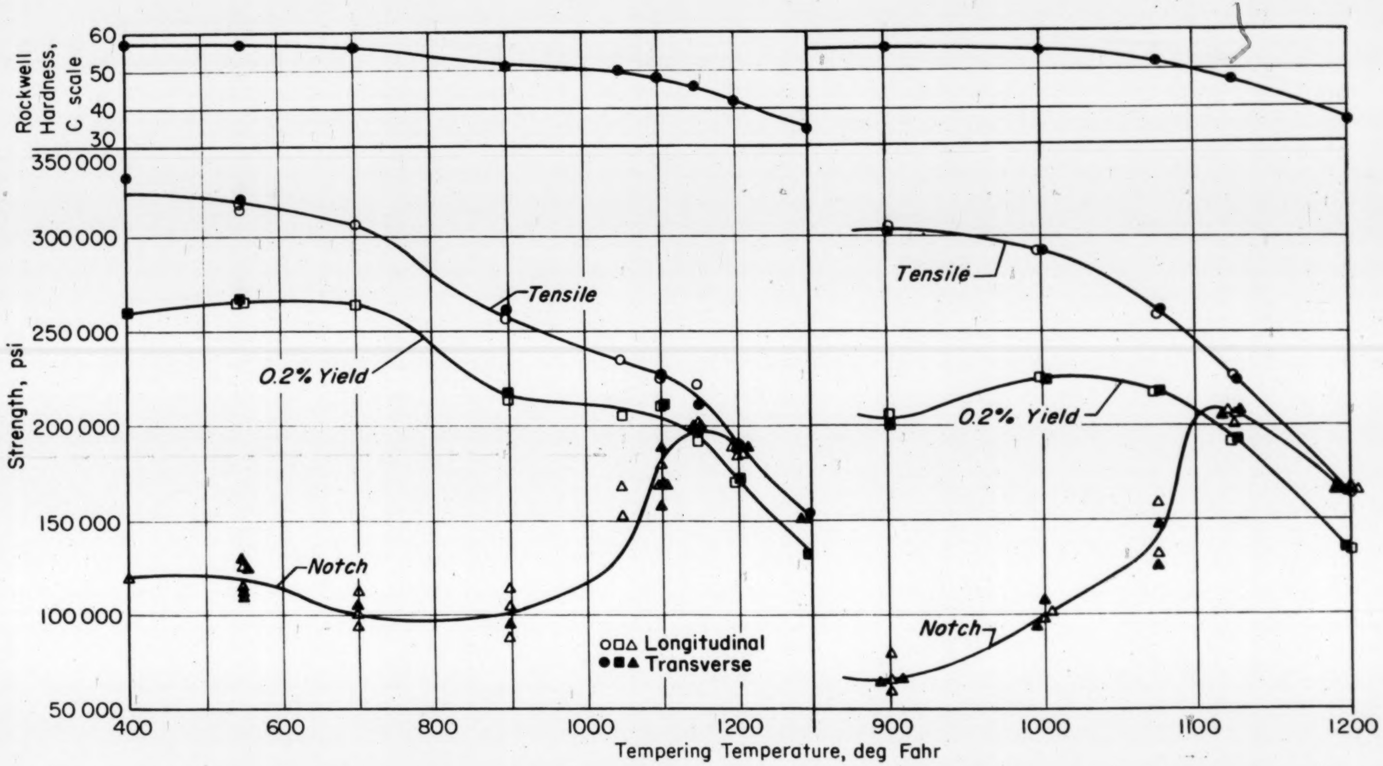
TEST RESULTS

Typical examples of the primary smooth and sharp-edge-notch data are shown in Fig. 2 for consumable electrode vacuum melted H-11 modified and 300M steels.

The variation in smooth and notch properties with tempering temperature[5] shown in Fig. 2 is typical of that re-

---

[5] Elongation values are not shown since they do not bear any definite relation to the notch sensitivity.

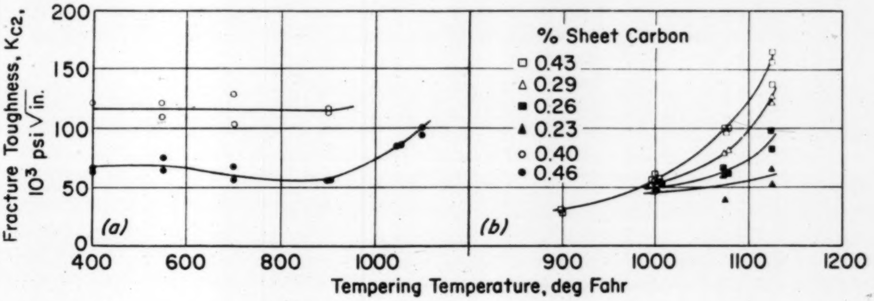


(*a*) 300M sheet steel, 0.45 per cent carbon.

(*b*) H-11 modified sheet steel, 0.35 per cent carbon.

FIG. 2.—Sharp-Edge-Notch and Smooth Tensile Strengths of Consumable Electrode Vacuum Melt 0.063-in. Sheet as a Function of Tempering Temperature.

ported previously (1) for heat treated steels and needs no detailed discussion here. Corresponding data for other carbon contents exhibit the same basic trends and comparable scatter. Maximum scatter is observed in that range of tempering temperatures corresponding to a rapid change in notch strength (that is, between 1000 and 1150 F for 300M and between 1050 and 1125 F for H-11 modified at the carbon levels represented in Fig. 2). This behavior is explained by the fact that these tempered conditions have transition temperatures at or near room temperature.



(*a*) 300M sheet steel at two carbon levels.

(*b*) H-11 modified sheet steel at various carbon levels.

FIG. 3.—Fracture Toughness of Air Melt 0.063-in. Sheet at Various Carbon Levels as a Function of Tempering Temperature.

The results of the fracture toughness (K_{c2}) analysis are shown in Figs. 3 (*a*) and (*b*) for both steels as a function of tempering temperature with the carbon content as parameter. The 300M steel notch specimens with carbon contents of 0.34 per cent and lower possessed net fracture stresses[6] above the yield strength and therefore could not be used for the purpose of fracture toughness calculation. For the same reason, the fracture toughness could not be determined for the higher tempered conditions of either alloy. Regarding fracture toughness, the authors wish to emphasize that the analysis for ink stain data is difficult in a 1 in. wide specimen. Because of the irregular nature of the stain near the termination point of the slow crack, rather arbitrary decisions must be made in determining the crack length. The effect of this difficulty would of course be reduced if the specimen were wider and consequently the total slow crack longer. In the present investigation, the analyses were made by only one person. The rather small scatter in Fig. 3 attests to his consistency in the procedure but does not necessarily indicate that others would obtain the same values. Furthermore, there is no experimental evidence available to the authors that proves K_c values determined from these edge notch specimens would be identical to those obtained from the 3 in. wide center slotted specimens frequently used for this purpose.

Measurements of the per cent shear in the fracture of notch specimens were made. However these results are not reported primarily because the authors were not able, in the case of the 300M alloy, to correlate such measurements with the indications of toughness derived from the strength values. Thus, the fracture appearance of this alloy at the 0.40 per cent carbon level is 100 per cent shear at the highest tempering temperatures when the notch strength is equal to the tensile strength. With decreasing tempering temperature, the per cent shear decreases with the notch strength, but returns to 100 per cent at the lowest tempering temperatures where the notch strength is considerably below the yield strength. This behavior has been illustrated previously (2).

---

[6] The net fracture stress is defined as the maximum load divided by the load-supporting area at the onset of rapid fracture (2).

DISCUSSION OF RESULTS

The following discussion relates to the effects of carbon content and vacuum remelting on the smooth strength and on the notch sensitivity of 300M and H-11 modified steels. The sharp-edge-notch tensile strength ratio[7] will be used as an indicator of the relative notch sensitivity for the various alloy conditions investigated. As mentioned previously, use is also made of the fracture toughness value K_{c2}. Consideration will be given to the variation in notch sensitivity or fracture toughness with the tensile strength. The yield strength might also

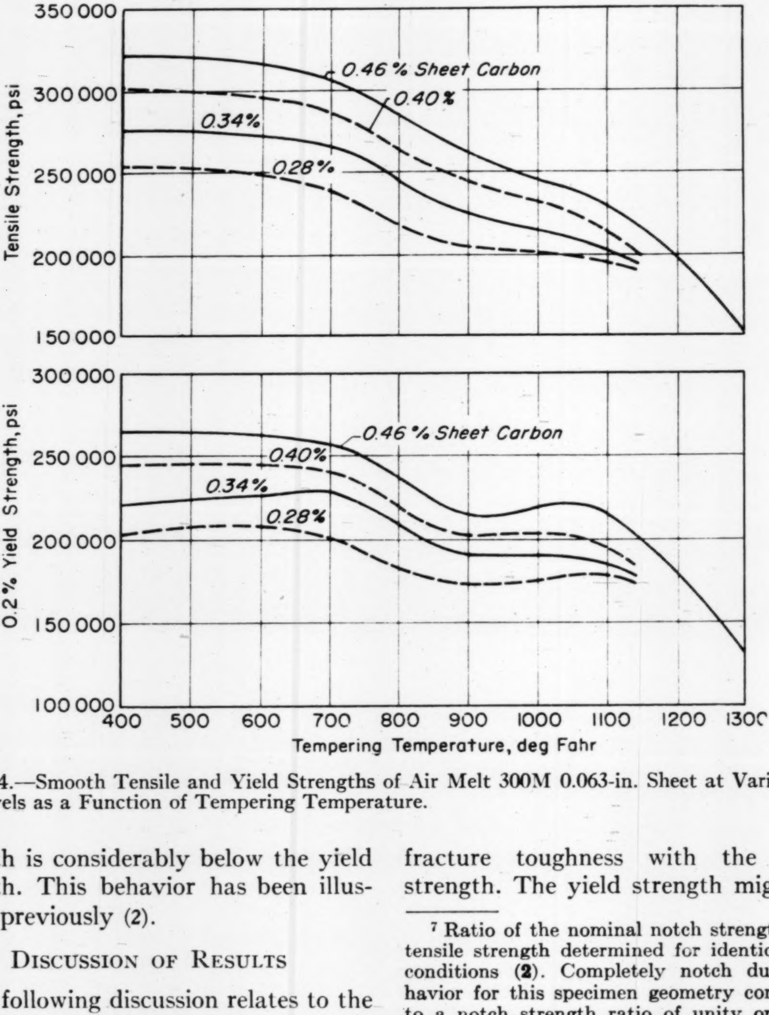


FIG. 4.—Smooth Tensile and Yield Strengths of Air Melt 300M 0.063-in. Sheet at Various Carbon Levels as a Function of Tempering Temperature.

---

[7] Ratio of the nominal notch strength to the tensile strength determined for identical metal conditions (2). Completely notch ductile behavior for this specimen geometry corresponds to a notch strength ratio of unity or slightly above and indicates an extremely tough metal condition. Values of the ratio below unity indicate the development of notch sensitivity; the lower the ratio, the higher the notch sensitivity.

be used as the independent variable. However, its use would complicate the analysis since it is frequently a double-valued function of the tempering temperature (see Figs. 4 and 5). Furthermore, it is influenced in a complex manner by residual stresses and is not a basic measure of the strength potential of the alloy. Nevertheless, the general effects revealed would not be altered by using the yield rather than the tensile strength, and a conversion can be made with the aid of Figs. 4 and 5.

Effect of Carbon Content and Melting Practice on Smooth Strength:

The smooth yield and tensile strength for various carbon levels of air melted 300M and H-11 modified alloys are shown in Figs. 4 and 5 as a function of the tempering temperature. The influence of tempering temperature and of carbon content on the smooth strength properties of such steels has been discussed previously (1,22). These steels exhibit the expected behavior in that the smooth strength is increased with increasing carbon content. This strengthening effect tends to decrease at the highest tempering temperatures.

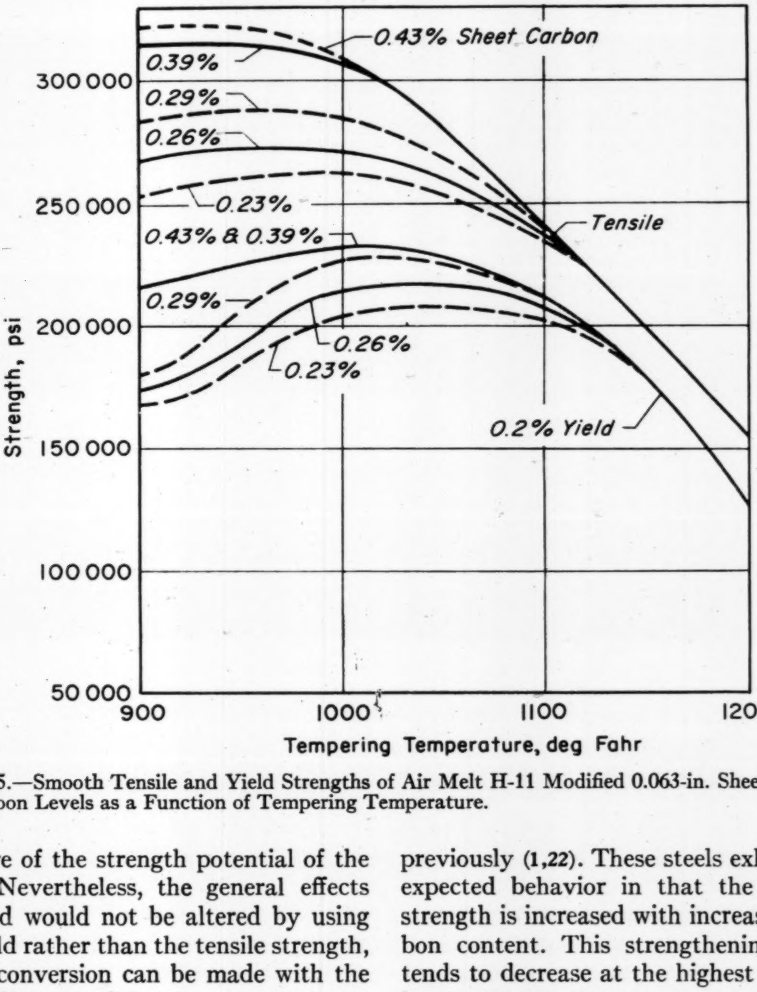


FIG. 5.—Smooth Tensile and Yield Strengths of Air Melt H-11 Modified 0.063-in. Sheet at Various Carbon Levels as a Function of Tempering Temperature.

The data may be analyzed for the effects of melting practice using the methods described in a later section concerning the influence of melting practice on notch sensitivity. Such an analysis reveals no difference between the smooth tensile properties of air and consumable electrode vacuum remelted material.

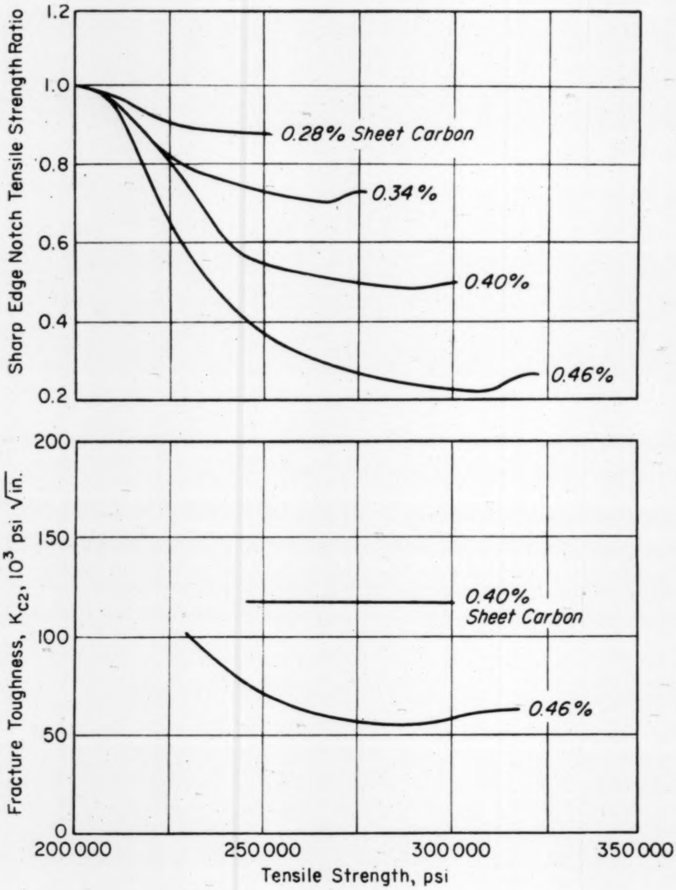


FIG. 6.—Sharp-Edge-Notch Tensile Strength Ratio and Fracture Toughness of Air Melt 300M 0.063-in. Sheet at Various Carbon Levels as a Function of Tensile Strength Level.

Effect of Carbon Content on Notch Sensitivity:

The sharp-edge-notch tensile strength ratio and the fracture toughness, K_{c2}, are shown as functions of tensile strength level in Figs. 6 and 7 for various carbon levels of air melted alloys 300M and H-11 modified, respectively. These curves were derived from plots of the primary data; examples of which were given in Fig. 2. The notch strength ratio and the fracture toughness curves exhibit parallel trends.

The influence of tensile strength level on the notch sensitivity of these types of steels has been discussed previously in detail (1). Notch sensitivity develops with increasing strength level in a similar manner at all carbon levels. However, the effect of strength level is somewhat different for the two steels. In the case of 300M alloy both the notch strength ratio and the fracture toughness decrease rapidly in a rather narrow range of

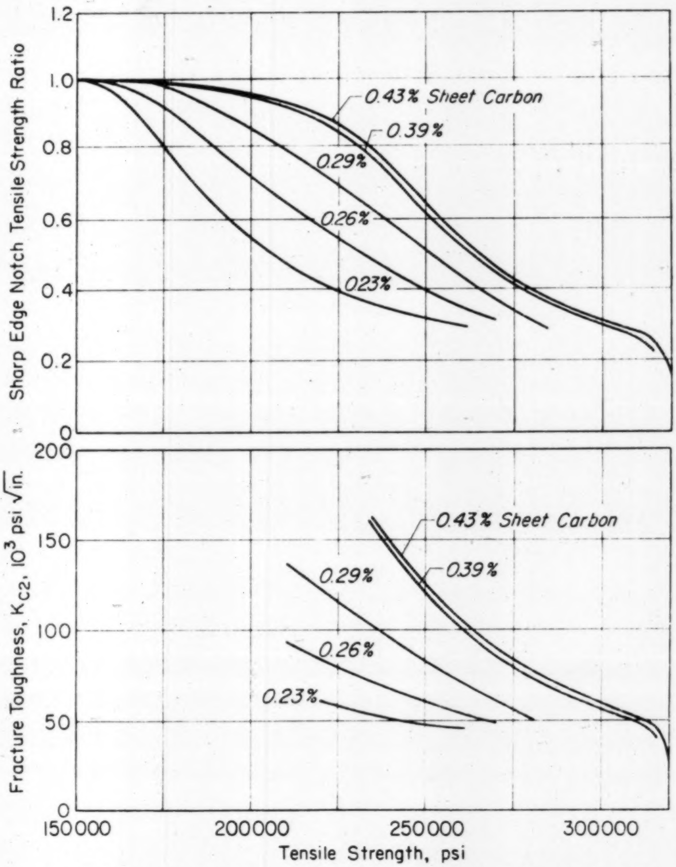


FIG. 7.—Sharp-Edge-Notch Tensile Strength Ratio and Fracture Toughness of Air Melt H-11 Modified 0.063-in. Sheet at Various Carbon Levels as a Function of Tensile Strength.

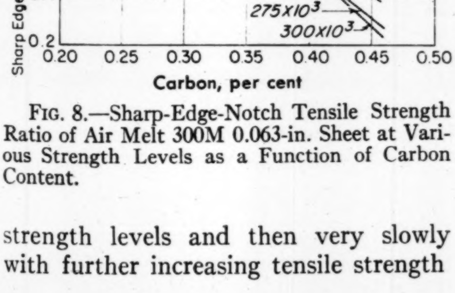


FIG. 8.—Sharp-Edge-Notch Tensile Strength Ratio of Air Melt 300M 0.063-in. Sheet at Various Strength Levels as a Function of Carbon Content.

strength levels and then very slowly with further increasing tensile strength At the highest strength levels, a shallow minimum is observed. In contrast, notch sensitivity in the H-11 modified steel develops more gradually with increasing strength level but increases continuously to the highest tensile strengths.

The influence of the carbon content can be clearly seen if the data in Figs. 6 and 7 are cross-plotted to show the notch strength ratio and the fracture toughness as a direct function of the carbon content with the tensile strength level as a parameter. Such representations are shown in Figs. 8 and 9. In the case of 300M steel, the fracture toughness is not shown since values are obtainable at only the two highest carbon levels.

According to these representations, distinctly different effects of carbon content are observed for the two steels. For 300M, fully notch ductile behavior is obtained at tensile strength levels less than about 200,000 psi for the entire range of carbon contents investigated. At higher strength levels, notch sensitivity increases continuously with increasing carbon content.[8] Generally, this increase is more rapid the higher the strength level. It will be noted that high notch sensitivity (notch strength ratios less than 0.4) characterizes the strongest conditions at high carbon contents. This basic effect of carbon content is similar to that reported for the notch strength of SAE 4300 steels by Klier (6) and for the impact properties of several low alloy steels by Schwartzbart and Sheehan (4,5).

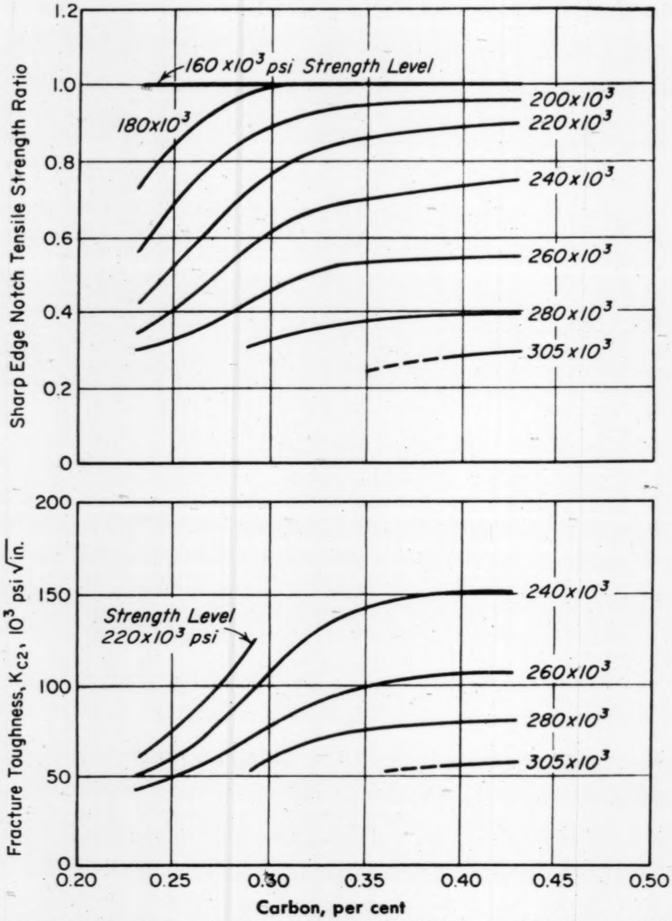
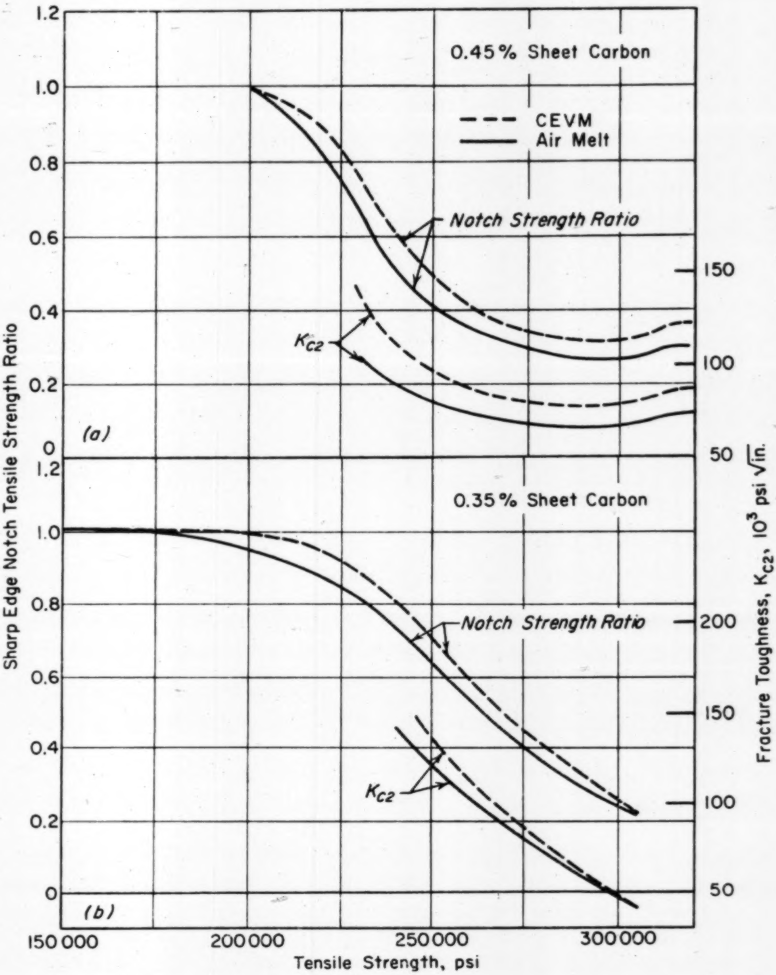


FIG. 9.—Sharp-Edge-Notch Tensile Strength Ratio and Fracture Toughness of Air Melt H-11 Modified 0.063-in. Sheet at Various Strength Levels as a Function of Carbon Content.

In the case of H-11 modified steel, the effect of carbon content is opposite to that observed in the 300M alloy. According to Fig. 9, fully notch ductile behavior is obtained at strength levels below about 160,000 psi for the range of carbon contents investigated. At higher strength levels, the notch sensitivity decreases with increasing carbon content. The authors have no explanation for this unexpected behavior. The carbon effect is most pronounced at strength levels below about 260,000 psi and fades out gradually at carbon contents above about 0.35 per cent. It is interesting to note that the highest strength conditions achieved at any given carbon level possess nearly equal and high notch sensitivity as indicated by notch strength ratios of 0.3 or below and fracture toughness values of about 50×10^3 psi $\sqrt{\text{in}}$.

---

[8] The curves for certain high strength levels will intersect, since the notch strength ratio is a double valued function of the strength level for this steel (see Fig. 6).



(*a*) 300M sheet steel, 0.45 per cent carbon.
(*b*) H-11 modified sheet steel, 0.35 per cent carbon.

Fig. 10.—Sharp-Edge-Notch Tensile Strength Ratio and Fracture Toughness as a Function of Tensile Strength Level for Air and Consumable Electrode Vacuum Melt 0.063-in. Sheet at the Same Carbon Levels.

This latter observation is more evident in Fig. 7.

In the preceding discussion attention has been directed to only one variable, namely, carbon content, which influences the properties of the steels investigated. It may be argued that other variables such as heat size, ingot size, or differences in composition other than carbon could influence the results. On the other hand, the continuous nature of the curves representing notch sensitivity as a function of carbon content and the definite family relationship between these curves at various strength levels strongly indicate carbon content was by far the most potent variable.

Effects of Melting Practice on Notch Sensitivity:

In order to determine the effects of consumable electrode vacuum remelting, it is necessary to compare air and vacuum melted steel at the same carbon level. This may be accomplished by interpolation of the curves given in Figs. 8 and 9 to yield the sharp-edge-notch tensile strength ratio and fracture toughness (K_{c2}) of air melted steel at the carbon level of the vacuum melted material.[9] The results of such an analysis are presented in Figs. 10(*a*) and (*b*), which show the notch strength ratio and fracture toughness as a function of strength level for 0.45 per cent carbon 300M and 0.35 per cent carbon H-11 modified, respectively. For both steels, only small improvements characterize the consumable electrode vacuum remelted material. These appear to be somewhat more pronounced in 300M than in H-11 modified. As might be expected, the effect fades out in both steels at low strength levels corresponding to notch ductile behavior. Furthermore, the effect of vacuum melting tends to disappear at the highest strength levels in the H-11 modified steel. These results are supported by recent data of Bernstein and Young (20) which indicates essentially identical fracture toughness values for air and consumable electrode vacuum melted 300M 0.080- and 0.220-in. sheet tested at 190,000 and 230,000 psi yield-strength levels.

PRACTICAL SIGNIFICANCE OF RESULTS

It is advantageous to discuss the significance of the results to both the alloy producer and the fabricator rather than to present a formal list of conclusions. In doing so, the authors make use of related data published by other investigators.

Regarding the influence of carbon content, it would appear that to obtain minimum notch sensitivity in 300M, the carbon content should be as low as possible consistent with the desired strength level. For example, at a tensile strength level of 275,000 psi (approximately 230,000 psi yield strength), the notch strength ratio is about 0.38 for the nominal carbon level of 0.43 per cent. Reducing the carbon content to 0.35 per cent increases the notch strength ratio at 275,000 psi to about 0.7. The corresponding fracture toughness of the 0.43 per cent carbon material is about 85×10^3 psi $\sqrt{\text{in.}}$ and is above 140×10^3 psi $\sqrt{\text{in.}}$[10] at the 0.35 per cent carbon level. These are very substantial effects and would correspond to a significant increase in the reliability of hardware. Similar effects could be expected in other low-alloy steels. This generality is suggested by the results obtained by Klier (6) for the edge notch strength of SAE 4300-V modified steels of different carbon contents. It is also supported by the impact data obtained by Schwartzbart and Sheehan (4,5) for a series of SAE compositions.

---

[9] It should be noted that a slight extrapolation of the 305,000 psi strength level curves for H-11 modified (Fig. 9) is required for this purpose. This extrapolation can introduce little error in the analysis because of the small variation in notch strength ratio and fracture toughness in this range of carbon contents.

[10] This is approximately the maximum value that can be measured at this yield strength level with the 1 in. wide specimen used in this investigation. The maximum value may be calculated according to Irwin (2) from the following relation: $K_m^2 = 0.38\sigma_y^2 w$, where σ_y is the yield strength and w is the specimen width.

For the 5 per cent chromium tool steel, H-11 modified, it would appear that the carbon content should not be lower than about 0.35 per cent to obtain minimum notch sensitivity at a given intermediate strength level. For example, at a strength level of 260,000 psi, the notch strength ratio increases from 0.3 at a carbon content of 0.23 per cent to 0.53 at a 0.35 per cent carbon level. Correspondingly, the fracture toughness increases from about 43×10^3 psi $\sqrt{\text{in.}}$ to about 100×10^3 psi $\sqrt{\text{in.}}$ While no data exist, it is assumed that at sufficiently high carbon levels the notch sensitivity would increase. If the alloy is to be used in its highest strength conditions, the benefit gained by increasing the carbon content is very small. The results reported here are in substantial agreement with those of Hamaker and Vater for the same basic alloy type. Further confirmation is supplied by a limited amount of data reported by Klier (6). Whether or not this behavior characterizes other tool steels is not known.

While the effects of carbon content vary with the alloy, the present results emphasize the fact that any steel development program should include the effects of varying carbon on the notch sensitivity. Furthermore, it should be emphasized that in order to obtain consistent results the sheet carbon should be measured since it may vary considerably from the melt carbon. In addition, unintentional nonuniform decarburization of the sheet must be avoided.

Regarding the effects of consumable electrode vacuum remelting, the results suggest that only rather small reductions in notch sensitivity of sheet are possible from remelting high cleanliness aircraft quality steel. Unfortunately it is not generally possible to derive definite conclusions from other published information, since in most cases it is not clear whether or not differences in sheet carbon existed between the air and vacuum melted alloys. However, some recent data by Bernstein and Young (20) for AMS 6434 appear to indicate improvement due to consumable electrode vacuum melting. Both 0.080 and 0.220 in. thick sheet were tested in the longitudinal and transverse directions at yield strength levels of 190,000 and 210,000 psi. Little difference was noted between air and vacuum melted material for longitudinal tests. Tests in the transverse direction revealed a very substantial improvement in the vacuum melted alloy. Since only minor differences in longitudinal properties were reported, it may be assumed that the air melt and the vacuum melt had the same carbon content. The cleanliness of both the air and vacuum melted material was considered to be exceptionally good.

Apparently the effects of consumable electrode vacuum remelting depend in part on the alloy composition as well as on other unknown factors. It may be possible to adapt the composition to take advantage of the vacuum remelting process. Furthermore, none of the present results is sufficient to establish clearly whether or not the uniformity of properties is improved by consumable electrode vacuum remelting. This possibility should be explored since improvements in this respect have been noted in the fatigue strength.

Acknowledgments:

The authors wish to acknowledge the cooperation of the Allegheny-Ludlum

Steel Corp., Vanadium Alloys Steel Corp., and the International Nickel Co. in making available the various steels used in this investigation. The authors appreciate the assistance of M. H. Jones of the Strength of Materials Branch, NASA Lewis Research Center, in interpretation of the ink stains. Also the authors wish to thank Miss Zita Mullen, Alloys and Composite Materials Branch, NASA Lewis Research Center, for performing the sheet carbon analyses.

APPENDIX

Several heats of each alloy (see Table I) exhibited an appreciable difference in carbon level between their melt and as-heat-treated sheet analyses (0.04 to 0.08 per cent carbon). To determine whether this difference was due to surface decarburization or to an apparent uniform carbon loss through the sheet's thickness, hardness studies were made of failed notch specimens heat treated at the highest tempering temperature.

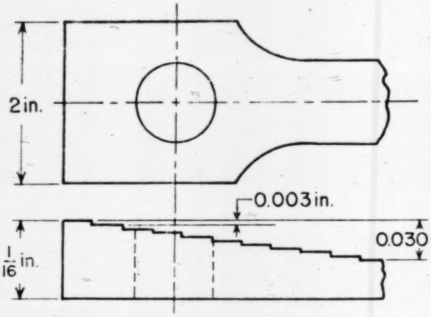


FIG. 11.—Specimen Step-Ground for Hardness Study.

Duplicate specimens of each material were surface ground to expose various planes through their thickness. Specimens were first ground flat on one side and then just "cleaned-up" on the other. Using very gentle grinding techniques, the "cleaned-up" side was step-ground as shown in Fig. 11.

At least three Rockwell readings were taken using the 30N scale on each step of the specimen. With the exception of 300M heats numbered 25-996, 25-997, and 25-998, no difference in hardness could be detected throughout the thickness. For these latter heats, a very mild hardness gradient was noted. This varied from specimen to specimen but never represented a difference greater than 3 points on the Rockwell 30N scale between surface and center. The indicated decarburization apparently had no noticeable effect on the notch sensitivity, as evidenced by the fact that no more scatter was observed in the strength values of these heats than in others showing no decarburization.

REFERENCES

(1) G. B. Espey, M. H. Jones, and W. F. Brown, Jr., "The Sharp Edge Notch Tensile Strength of Several High-Strength Steel Sheet Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 837 (1959).

(2) "Fracture Testing of High Strength Sheet Materials: A Report of a Special ASTM Committee," ASTM BULLETIN, No. 243, Jan. 1960, p. 29; No. 244, Feb., 1960, p. 18.

(3) G. B. Espey, M. H. Jones, and W. F. Brown, Jr., "Sharp-Edge-Notch Tensile Characteristics of Several High-Strength Titanium Sheet Alloys at Room and Cryogenic Temperatures," Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials, Am. Soc. Testing Mats., p. 74 (1960). (Issued as separate publication *ASTM STP No. 287*)

(4) H. Schwartzbart and J. P. Sheehan, "Effect of Specimen Size on Notched Bar Impact Properties of Quenched and Tempered Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 939 (1954).

(5) J. P. Sheehan and H. Schwartzbart, "Impact Properties of Quenched and Tempered Alloy Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 483 (1956).

(6) E. P. Klier, "The Effect of Carbon Content on Notch Properties of 43XX-Vanadium Modified and 5 per cent Chromium Sheet Steels," Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials, Am. Soc. Testing Mats., p. 196 (1960). (Issued as separate publication *ASTM STP No. 287.*)

(7) P. Payson and A. E. Nehrenberg, "A New

Steel Features High Strength and High Toughness," *Iron Age*, Vol. 162, Oct. 21, p. 64, and Oct. 28, p. 74 (1948).

(8) G. Sachs, G. S. Sangdahl, and W. F. Brown, Jr., "New Notes on High Strength Heat Treated Steels," *Iron Age*, Nov. 23, p. 59, and Nov. 30, p. 76 (1950).

(9) J. C. Hamaker, Jr., and E. J. Vater, "Carbon-Strength Relationships in 5 per cent Chromium Ultra High Strength Steels," see p. 691, this publication.

(10) "Vacuum Remelting of Super Alloys and Steels by the Consumable Electrode Process," in "Arcs in Inert Atmospheres and Vacuum," John Wiley & Sons, New York, N. Y. (1956).

(11) W. W. Dyrkacz and F. E. Reynolds, "Vacuum Melted Alloys for Forged Parts," *Product Engineering*, Vol. 28, p. 143, Feb. 1957.

(12) P. H. Frith, "Fatigue Tests on Rolled Alloy Steels Made in Electric and Open Hearth Furnaces," Special Report No. 50, Iron & Steel Institute, London (1954).

(13) G. Sachs, R. Sell, and W. F. Brown, Jr., "Tension, Compression, and Fatigue Properties of Several Steels for Aircraft Bearing Applications," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 635 (1959).

(14) J. T. Ransom, "The Effect of Inclusions on the Fatigue Strength of SAE 4340 Steels," *Transactions*, Am. Soc. Metals, Vol. 46, p. 1254 (1954).

(15) S. Cvikevich, "Mechanical Properties of AISI 4340 Steel Improved by Consumable Electrode Vacuum Remelting," Technical Report No. WAL TR 310.1/1, Watertown Arsenal Laboratories, Sept. 1959.

(16) G. Zima, "Some Effects of Melting Atmosphere Variation on the Properties of Inco Ultra and Super Ultra High Strength Steels," International Nickel Co., New York, N. Y., Oct. 1958.

(17) M. Gensamer, "Study of the Effects of Vacuum Melting on 550°F Tempering Embrittlement," WADC Technical Report 57-85, 1957.

(18) P. Blanchard and A. R. Troiano, "Delayed Failure and Notch Tensile Properties of a Vacuum Melted 4340 Steel," WADC Technical Note 58-176, Sept. 1958.

(19) R. H. Raring and J. A. Rinebolt, "Static Fatigue Strength of High Strength Steel," *Transactions*, Am. Soc. Metals, Vol. 58, p. 198 (1956).

(20) H. Bernstein and G. C. Young, "Toughness Evaluation of Air and Consutrode Melted Tricent (300M) and AMS 6434," NRL Project 62R1905 Technical Memorandum No. 117, Mar. 14, 1960, U.S. Naval Weapons Plant, Washington 25, D.C.

(21) J. L. March, W. J. Ruprecht, and G. Reed, "Machining of Notched Tension Test Specimens," ASTM BULLETIN, No. 244, Feb. 1960, p. 52.

(22) G. Sachs, "Survey of Low Alloy Steels Heat Treated to High Strength Levels. Pt. 4: High Strength Steels and Their General Static Properties," WADC Report 53-254, 1953.

DISCUSSION

MESSRS. G. A. ROBERTS[1] AND J. C. HAMAKER,[1] JR. (*presented in written form*).—The authors have again made excellent use of their edge notch test to study the effect of carbon content and vacuum melting on the sharp-notch characteristics of two high-strength steels. The rather unexpected increase in notch strength of H-11 modified steel with increasing rather than decreasing carbon content has been confirmed in our own and other investigations, and offers an intriguing and potentially valuable guide for future alloy development. For ultimate strength capabilities beyond 300,000 psi, higher carbon contents beyond the conventional 0.30 to 0.40 per cent range normally found in structural steels are essential. Apparently, the higher alloy content of H-11 modified steel permits it to carry this increased carbon without detriment to the notch strength; in fact, the notch strength is improved to a measurable degree. Preliminary results on higher alloy compositions than H-11 modified further confirm this trend of greater carbon carrying and, thereby, strengthening ability with increased alloy.

The authors' data confirmed the indications of their previous studies, and those of other investigators, that with extremely sharp notches high-strength steels generally show about equivalent increases in notch sensitivity with increasing heat-treated strength level. When the various edge notch, center notch, and center crack test data are plotted against heat-treated strength level, the values tend to fall within a single band generally within the statistical accuracy of the particular test. Some tests may show slight advantages for one steel or another, depending on the specimen geometry and test conditions employed, but the increasing volume of data is irrevocably pointing toward the conclusion that no single composition will defy the trend to increasing sharp notch sensitivity as the strength level is increased. For example, such a comparison plot of the data in the paper at a given carbon level shows slight notch ratio advantages for one steel and then the other at various strength levels, but, in all cases, the ratios are within 0.05 to 0.10 of each other and certainly within the scatter that might be expected with heat to heat variations, as well as specimen and test variations.

However, this need not be as discouraging as it seems because in engineering design there appears to be a complete range of notch sharpness criteria besides the extremely sharp notch concentration factors studied here by the authors. Depending on the design, notches from the sharp $K = 12$ to 15 studied here, to the range of 4 to 6 used in many engineering applications, down to $K = 1$ (smooth bar) become important. For example, recent developments in missile case manufacture have indicated that a K of only 4 to 6 may be significant to well-designed and fabricated vessels.[2] In this notch sharpness range, results from several facilities indicate that modified H-11 retains a favorable notch strength ratio of

---

[1] Vice-President—Technology, and Director of Research, respectively, Vanadium-Alloys Steel Co., Latrobe, Pa.

[2] Rocket Motor Case Symposium, Pratt & Whitney Aircraft Co., Hartford, Conn., Sept. 29–30, 1959.

0.95 to 1.00 to ultimate strength levels of 280,000 to 300,000 psi.[2-4] Furthermore, at notch concentration factors of 4 to 6, the transition with test temperature occurs below −100 F on a notch tension test[2] and below −320 F on the V-notch Charpy impact test.[5] Preliminary results also indicate a somewhat greater advantage from consumable electrode vacuum melting than was found with the extremely sharp notches. Proceeding still further to $K = 1$ (smooth bar), consumable electrode vacuum melting has provided tremendous improvements of 100 to 300 per cent in the transverse ductility of billets for air frames and landing gear in sections of 7 to 16 in. square.

The improved methods for missile case manufacture have further indicated that a notch length of only 0.010 in. is critical to their design. Calculating from this figure, we obtain a $\mathcal{G}_c$ requirement of only 35, whereas the sharp-notch testing programs have generally assumed that a $\mathcal{G}_c$ of 600 to 1000 would be necessary for reliability in rocket cases—again, an indication that we may be becoming unduly alarmed by extremely sharp-notch test results that are not necessarily applicable to engineering design and manufacture.

With the wealth of very excellent notch data that have been developed in

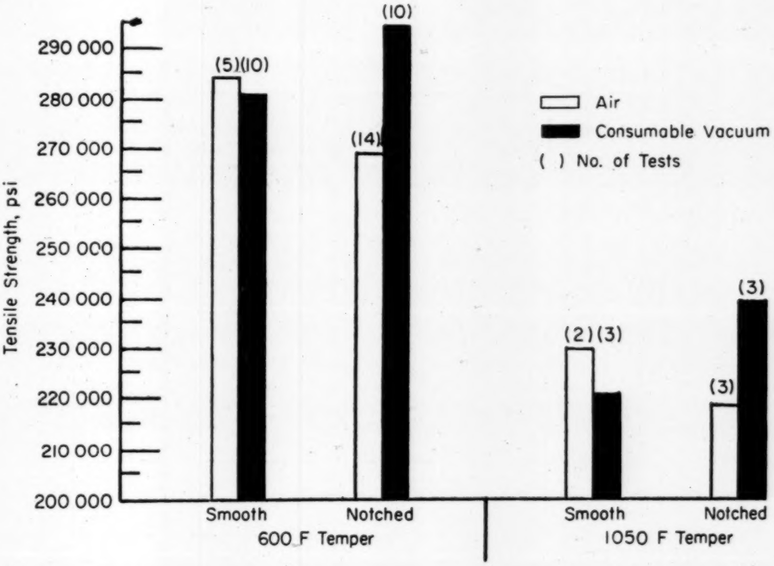


FIG. 12.—Smooth and Notch Tensile Properties of 300M Alloy Air Melt Versus Consumable Vacuum Melt.

Sheet specifications 0.500 in. wide by 0.080 in. thick, 0.005 in. root radius notch, $K_t = 5$ (ASTM Methods E 8 – 57 T). Heat treatment 1700 F—Fce, cool to 1600 F, air quench, double temper at given temperature.

---

[3] G. Sachs, J. Sessler, K. Grewal, and B. Weiss, "Investigation of Stress Concentrations and the Cracking Tendency of Heat-Treated Titanium and Steel Sheet Alloys," Buaer Contract NOas 58-855c, *Quarterly Progress Report 5*, Oct., 1959.

[4] R. Freymeyer, J. Brown, and L. Gilbert, "Evaluation of the Notch Sensitivity and Strength Under Biaxial Stress for Air Melted Ultra-High-Strength Steels," *Report No. M-1724*, Part I, Aerojet-General Corp., May, 1959.

[5] J. C. Hamaker, Jr., and E. J. Vater, "Carbon-Strength Relationships in 5 per cent Chromium Ultra-High-Strength Steels," see p. 691, this publication.

the authors' programs, it would appear extremely important to undertake additional programs to establish the relationship between various notch strength ratios, $\mathcal{G}_c$ factors, etc., and the performance and reliability of actual engineering structures. There are currently many aircraft structures and attachments flying with full reliability after exhaustive test programs, at strength levels considerably above those recommended by the sharp-notch testing results.

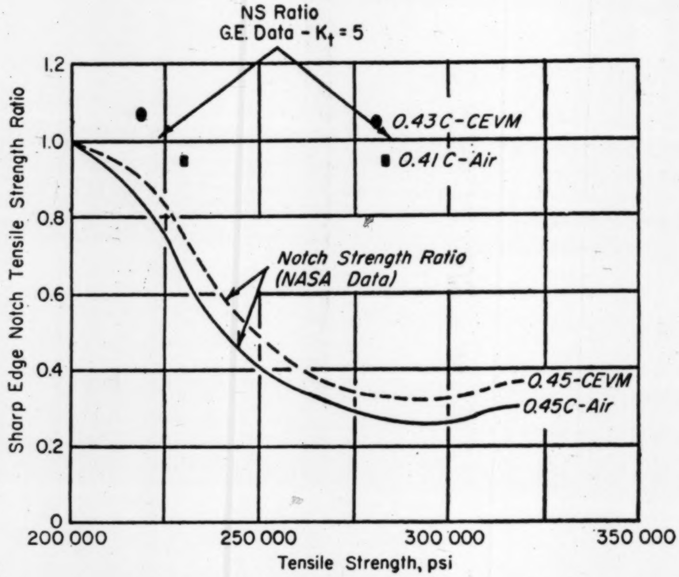


FIG. 13.—Smooth and Notch Tensile Properties of 300M Alloy Air Melt Versus Consumable Vacuum Melt (General Electric Co.). Data plotted against authors' data in Fig. 10(*a*).

MR. W. W. DYRKACZ[6] (*presented in written form*).—300M alloy sheet was evaluated by the Applied Research Metallurgical Laboratory of the General Electric Co., at Evendale, Ohio, for their Rocket Engine Section.[7] The program consisted of evaluating both air-melted and consumable electrode vacuum-melted 0.080 in. thick sheet. The air-melted heat contained 0.41 per cent carbon and was exceptionally clean. Two consumable vacuum-melted heats of 0.42 and 0.43 per cent carbon were tested. The edge notch tension tests had a 0.150 in. deep notch, 60 deg included angle with a 0.005-in. root radius in a 0.500 in. wide specimen to give a theoretical stress concentration factor of $K_t = 5$ which they believed would approximate the maximum stress concentration that would be expected in properly designed and fabricated rocket cases.

A comparison of the smooth and notched tensile strengths is given in the accompanying Fig. 12. The lower tensile strength for the consumable vacuum-melted material at the comparable carbon levels is abnormal, and we have no explanation for it at this time. However, these data are presented as a matter of general interest. The numbers in parentheses indicate the number of tests performed for each condition and mean values are shown in Fig. 12.

---

[6] Manager of Quality, Allegheny Ludlum Steel Corp., Watervliet, N. Y.

[7] Private communication from A. M. Krainness, General Electric Co.

It is of further interest to plot these data on Fig. 10(*a*) of the authors' paper, which is shown in the accompanying Fig. 13. The consumable electrode vacuum-melted material again has a higher notch strength ratio in these tests at the $K_t = 5$ condition. These data show substantially good agreement with data reported by Sachs and Sessler[8] at the same stress concentration level wherein the latter tested material from the same heat of 300M as did the authors.

It is to be noted that these data presented by both the authors and General Electric Co. were on exceptionally clean air melts. In translating these data into actual hardware, the problem arises of consistently procuring material of high quality. Therefore, it may be appropriate to review briefly the microcleanliness specification requirements for ultra-high-strength steels which indirectly reflect the availability on a day-to-day and heat-to-heat basis. Table III shows a comparison of typical microcleanliness specifications to which both air-melted and consumable electrode vacuum-melted steels are being currently purchased. It can be readily seen that most consistent cleanliness will be available in the consumable vacuum-melted product. In addition, similar quality differences are reflected by the Aeronautical Material Specifications for Magnetic Particle Inspection—AMS 2300 for consumable vacuum arc-melted and AMS 2301 for air-melted steels.

TABLE III.—TYPICAL MICROCLEANLINESS REQUIREMENTS OF HIGH-STRENGTH STEELS.

Worst Field Allowed (ASTM Recommended Practice E 45 – 51)[a] Method A Ratings

Type of Melt	Rating	A	B	C	D
Air	thin	2	2	2	3
	heavy	2	2	2	2
Consumable electrode vacuum arc	thin	1½	1	½	1½
	heavy	1	0	0	1

[a] Recommended Practice for Determining the Inclusion Content of Steel (E 45 – 51), 1958 Book of ASTM Standards, Part 3.

Secondly, the authors point out the importance of carbon content. A large number of the missile parts require the use of large slabs for sheet rolling and billets for forged rings. The carbon variation within large, conventionally cast ingots may be as high as 5 points, due to segregation. Since smaller cross-section cast electrodes are used to produce consumable electrode melted ingots of comparable size, there is less segregation initially. In addition, on consumable remelting, segregation is minimized through the rapid cooling occurring in the copper, water-cooled crucible. When the necessity for "zeroing-in" on carbon content develops, consumable electrode vacuum melting will offer a further quality advantage in addition to the present cleanliness advantage.

Therefore, it is evident that during the selection of materials for rocket motor cases, the producibility engineer should consider the reliability of material quality and availability at the same time that the design engineer is analyzing such mechanical property data as those presented by the authors.

MR. FRANCIS PALL[9] (*presented in written form*).—It may be of interest to discuss the possible effects of a slight increase in specimen width, W, over the 1-in. value used in this investigation. Since the thickness B was constant at 0.063 in., this would mean a width to thickness ratio W/B of more than 16.

---

[8] George Sachs and John G. Sessler, "Effect of Stress Concentration on Tensile Strength of Titanium and Steel Alloy Sheet at Various Temperatures," Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials, *ASTM STP No. 287*, Am. Soc. Testing Mats, p. 122 (1960).

[9] Assistant Research Engineer, Boeing Airplane Co., Aero-Space Division, Seattle, Wash.

Such an increase might tie pressure vessel brittleness closer to sharp notch sensitivity without appreciably affecting specimen economy through an increased W.

It is convenient to call a thin-walled pressure vessel tough when the calculated burst strength, F_{TB}, exceeds the tensile yield strength, F_{TY}. This implies that the stress which has caused yielding in the vessel must have been approximately equal to that which has caused yielding in the uniaxial tension specimen. Or, in terms of the maximum shear criterion, at yielding the maximum shear stress in vessel and uniaxial specimen were practically equal. But for the shear criterion to apply, it is necessary to have ample shear prior to maximum load. Ample shear may mean $P = 100$ per cent shear lips at the origin of fracture, as defined in reference (2) of the paper. However, according to G. R. Irwin,[10] even at $P = 80$ per cent a moderate safety margin gives some assurance of adequate pressure vessel toughness.

From Fig. 3 in reference (2) of the paper the beta parameter equals 8, when P equals approximately 83 per cent. Then, in Eq 1 of reference (2) of the paper the beta parameter should equal or be larger than 8:

$$K_c^2/B\,F_{TY}^2 = 8 \ldots\ldots\ldots\ldots (1)$$

The specimen, in accordance with this view, must be capable of measuring a toughness which is large enough to make this equation equal to at least 8. The maximum measurable toughness, K_c (max), in terms of W, from reference (2) of the paper is:

$$K_c^2\,(\text{max}) = (0.38)F_{TY}^2\,W \ldots\ldots (2)$$

From Eqs 1 and 2 the width of the specimen which will measure this larger toughness, for $B = 0.063$ in., is:

$$W = (0.063)(8)/0.38 = 1.33 \text{ in.}$$

The maximum measurable toughness, K_c (max), by this specimen would represent in principle the condition at which F_{TB} exceeds F_{TY} rather than the limiting condition under which a $2B$ long, essentially plane, stress crack is just arrested.

Then, as the actual toughness of the material, K_c, is improved, through better tempering, heat treatment, remelting, etc., K_c would increase and approach K_c (max) in the same manner as F_{TB}, or notch strength, F_{TN}, would increase, approach, or exceed F_{TY}.

This could facilitate correlations the industry might want to develop between such ratios as K_c/K_c (max), F_{TB}/F_{TY} or F_{TN}/F_{TY}.

However, in specific instances, as the authors point out, the use of P, or beta, in Eq 1 leads to contradictions. For example, for 300M, 0.40 per cent carbon, air melt, at tempers 500, 600 and 700 F, from Fig. 4 of the paper, K_{c2} is constant at 120,000 psi $\sqrt{\text{in.}}$ F_{TY} in this range is approximately 246,000 psi. These in Eq 1 give a beta value of 3.78, which is between one half and one third of what is necessary for the shear criterion to apply since $(8)(1/2.11) = 3.78$ or $(10)(1/2.64) = 3.78$. On the average then, beta should be $(2.64 + 2.11)/2 = 2.4$ times larger. But as it is, the measured K_{c2}^2 is too small by 2.4 and K_{c2} by $\sqrt{2.4} = 1.55$. Then, if the ratios K_{c2}/K_{c2} (max) and F_{TN}/F_{TY} are approximately proportional, the actual F_{TN}/F_{TY} too will be low by 1.55.

Indeed, two 6.5-in. diameter cylindrical tanks of this material tempered at 500 F to 300,000 psi ultimate strength have shown an average F_{TB}/F_{TY} of 1.22, indicating that the shear criterion is applicable, beta should have been between 8 and 10, but the corresponding F_{TN}/F_{TY} is only 0.62. At 600 F temper the average of three burst tests is $F_{TB}/F_{TY} = 1.10$. The corresponding F_{TN}/F_{TY} is 0.56. At 700 F temper the average of two burst tests gives $F_{TN}/F_{TY} = 0.9$, with F_{TN}/F_{TY} equaling 0.53.

---

[10] Private communication, George Irwin Mechanics Division, U. S. Naval Research Laboratory, Washington, D. C.

If the F_{TN}/F_{TY} values were multiplied by 1.55 they fall in line with the burst ratios F_{TB}/F_{TY}.

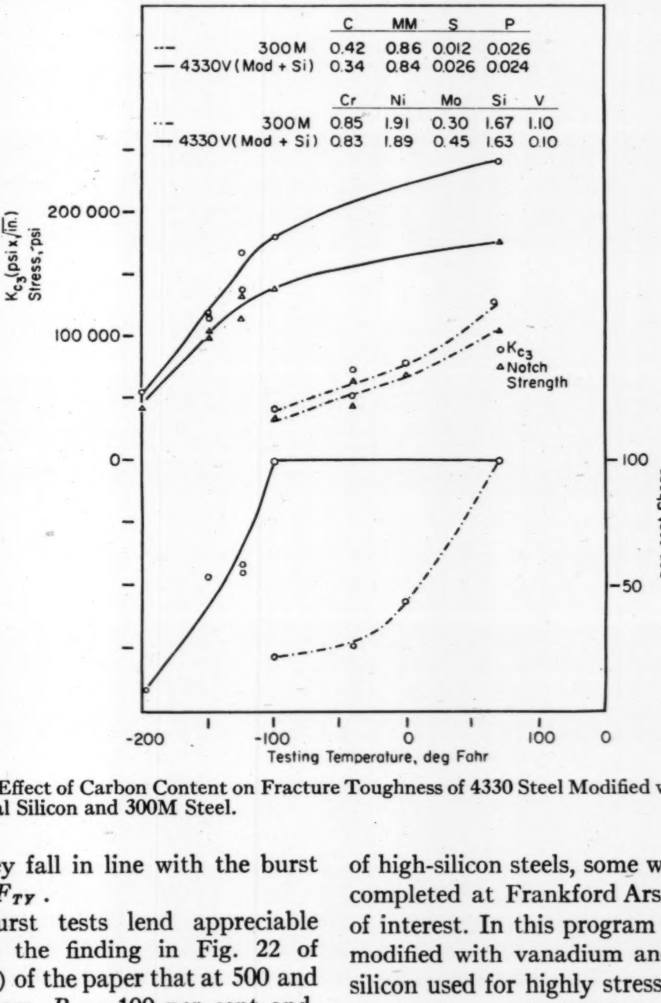


FIG. 14.—Effect of Carbon Content on Fracture Toughness of 4330 Steel Modified with Vanadium and Additional Silicon and 300M Steel.

These burst tests lend appreciable support to the finding in Fig. 22 of reference (2) of the paper that at 500 and 600 F tempers, $P = 100$ per cent and, just over 700 F temper, P decreases. It is probably worthwhile to note that a slight increase in the notch radius to about 0.005 in. could cause a 1.55 fold increase in F_{TN}/F_{TY}. This would not alter the general form of the stress equations at the root of the notch nor the fracture appearance index P.

MR. C. M. CARMAN[11] (*presented in written form*).—In regard to the effect of carbon content on the fracture toughness of high-silicon steels, some work recently completed at Frankford Arsenal may be of interest. In this program a 4330 steel modified with vanadium and additional silicon used for highly stressed ordnance components and 300M steel are compared by testing over a range of subzero temperature as originally described by

---

[11] Metallurgist, Frankford Arsenal, Philadelphia, Pa.

Srawley and Beachem.[12] The 3 by 12-in. centrally notched sheet specimen was used for those experiments. All specimens were approximately 0.080 in. thick. The results are shown in the accompanying Fig. 14.

In this figure, K_{c3} fracture toughness, notch strength, and per cent shear in the fast fracture are plotted as a function of testing temperature. The effect of reducing the carbon content is quite apparent. The transition from 100 to 50 per cent shear in the fast fracture occurs at +10 F for the 300M steel and at −140 F for the modified 4330 steel. At a testing temperature of −150 F the K_{c3} fracture toughness value for the modified 4330 steel is the same as that for the 300M steel at room temperature.

MR. E. P. KLIER[13] (*presented in written form*).—The authors have presented many new and interesting data for the evaluation of high-strength sheet metals, while they have also advanced perhaps the first relatively complete set of data for the K_c method of analysis of fracture toughness. From the fact that not all specimens failed in a manner allowing unambiguous measurement of K_c, two questions arise. On the one hand, is the best structural performance to be realized for the present materials for those conditions which allow the measurement of K_c or not? In our work it has been proposed that best structural performance may be realized for heat-treated conditions under which K_c is perhaps an ambiguous measurement. The second question pertains to the general interchangeability of K_c measurements.

Fundamentally, resolution of this question requires a full understanding of the stress system associated with different stress raisers and a full understanding of how different stress patterns influence fracture behavior. While the needed general knowledge both at the theoretical and experimental levels does not at present exist, specific problems can be approached by the use of specific tests. The results of fracture toughness tests on related specimens then should be reasonably interchangeable. On this basis the data obtained by the authors should be comparable with the data we have obtained on related steels.

For example, the 300M steels may be considered as related to the 43XX-V modified steels which we have studied, while the H-11 modified steels are of the group we have designated as 5 per cent chromium steels. Cross-comparison of the indicated sets of data, therefore, would seem to be legitimate. It is our belief that when the indicated comparisons are made, the authors' data are in essential agreement with our data on a quantitative basis. On a qualitative basis the trends established for the effects of carbon content in the two studies are in full agreement.

MESSRS. P. R. BORNEMAN[14] AND T. J. MCCAFFREY[14] (*presented in written form*).—The authors are to be complimented for recognizing the need for investigating the effects of chemical analysis and particularly carbon content upon the notch behavior of high-strength steels. Of particular interest is the behavior of the modified H-11 steel sheet in exhibiting improvement in notch strength with increased carbon content, while the reverse of this effect is found in 300M steel sheet. The explanation for this effect is probably found in the microstructural differences between the heat-treated structures of the two analyses, the modified H-11 material having appreciable alloy carbide in its structure

---

[12] J. E. Srawley and C. D. Beachem, "Crack Propagation Tests of Some High-Strength Sheet Steels," *NRL Report 5263*, U. S. Naval Research Laboratory, Jan. 10, 1959.

[13] Professor, The Catholic University of America, Washington, D. C.

[14] Tool and Die Research Section, Allegheny Ludlum Steel Corp., Pittsburgh, Pa.

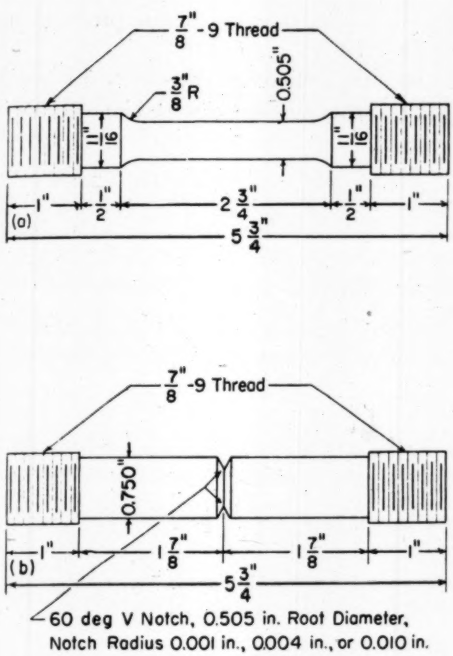

7/8"-9 Thread
3/8" R
0.505"
11/16"
11/16"
1"
1/2"
2 3/4"
1/2"
1"
(a)
5 3/4"
7/8"-9 Thread
0.750"
1"
1 7/8"
1 7/8"
1"
(b)
5 3/4"
60 deg V Notch, 0.505 in. Root Diameter,
Notch Radius 0.001 in., 0.004 in., or 0.010 in.

FIG. 15.—Smooth and Notched Tension Specimens.

(*a*) Unnotched tension specimen.
(*b*) Notched tension specimen.

while little alloy carbide is found in the 300M material. An investigation of the distribution of carbon between carbide and matrix martensite may yield a clue to the differences in the effect of carbon content on the notch strength of different alloys.

It is unfortunate that sufficient material was not available to investigate fully the effect of melting practice on notch strength in both the longitudinal and transverse directions and that a rigorous evaluation of cleanliness in these materials was not completed. A number of fabricators have reported improved formability resulting from consumable electrode vacuum melting with a resulting decrease in the incidence of crack formation during fabrication. This effect is attributed to the improvements in cleanliness, alloy segregation, and gas content known to result from consumable electrode vacuum-melting practice.

Although the magnitude of improvement in notch strength of these sheet

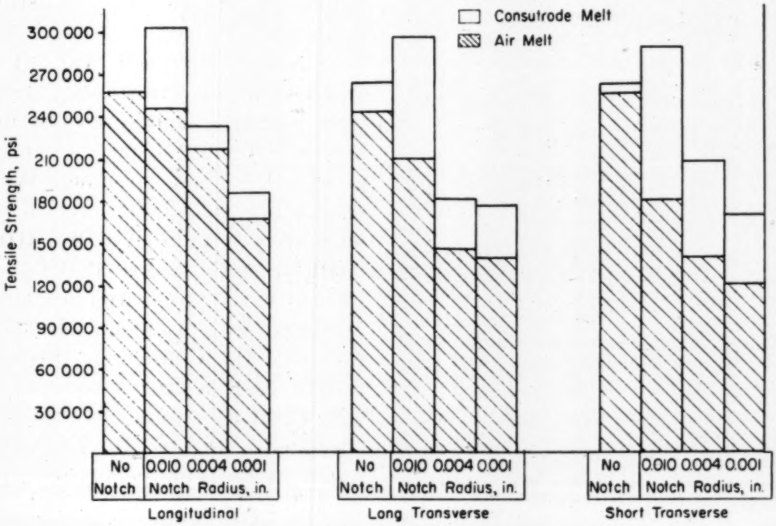


FIG. 16.—Effect of Testing Direction and Melt Practice on Notched and Unnotched Tensile Strength of Potomac A at 260,000 psi Tensile Strength Level.

materials resulting from consumable electrode vacuum-melting was found to be small, the consistency of this improvement would seem to be most significant.

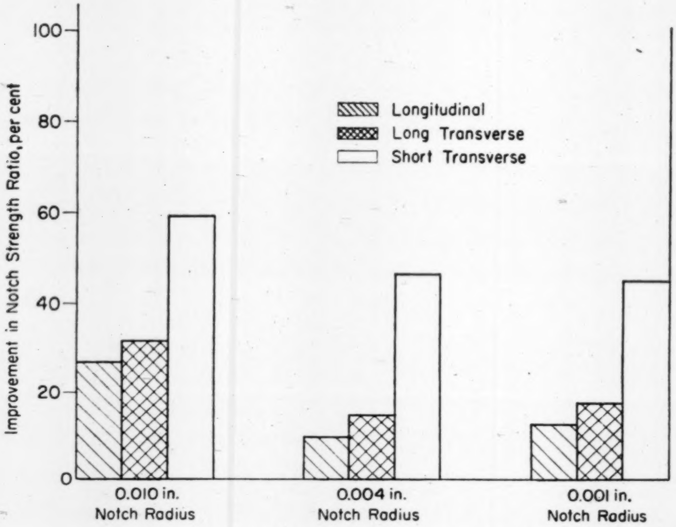


Fig. 17.—Percentage of Improvement in Notch Strength Ratio Realized from Consutrode Melting of Potomac A.

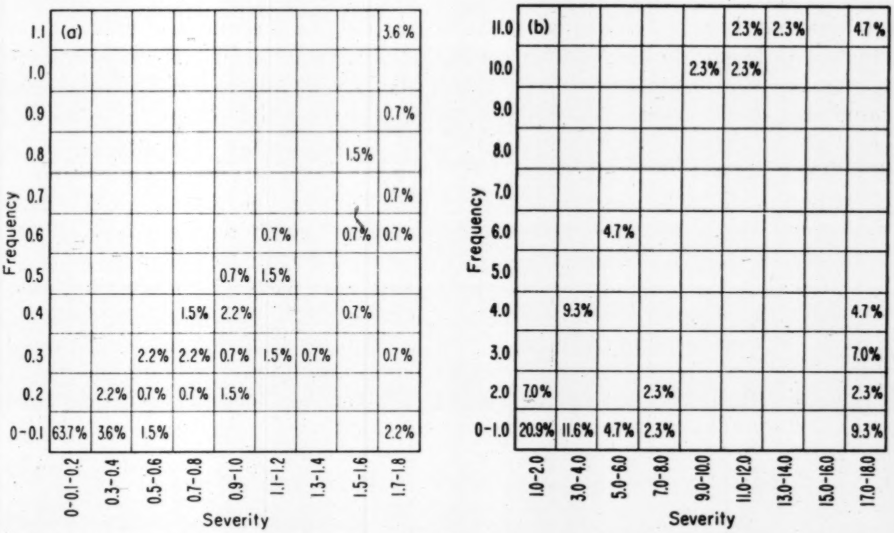
TABLE IV.—AVERAGE NOTCHED STRENGTH RATIOS.

Notch Radius, in.	K_t Factor[a]	Longitudinal		Long Transverse		Short Transverse	
		Air Melt	Consutrode Melt	Air Melt	Consutrode[b] Melt	Air Melt	Consutrode Melt
0.010....	4.7	0.93	1.18	0.86	1.13	0.69	1.10
0.004....	7.0	0.82	0.90	0.60	0.69	0.54	0.79
0.001....	13.7	0.64	0.72	0.57	0.67	0.45	0.65

[a] Based on a major diameter of 0.750 in. and a root diameter of 0.505 in. or d/D ratio of 0.672.

If notch strength is considered as a specific case of ductility measurement, the significance of this improvement increases, since it is known that ductility effects in martensitic steels are obscured when large amounts of work are performed, as is required in the production of sheet.

Data obtained by Allegheny Ludlum Steel Corp. on modified H-11 steel (Potomac A) in slab form illustrate the superiority in notch strength ratio of consumable electrode vacuum-melted material in all directions. These data were obtained from 6 by 12 in. slabs using the round test specimens illustrated in Fig. 15. These specimens were heat treated to the 260,000 psi tensile strength level. Notch radii of 0.010, 0.004 and 0.001 in. were evaluated. Of particular interest is the notch strengthening effect exhibited by specimens with 0.010 in. radius of the Consutrode melted material in each testing direction, whereas this effect is absent in the air-melted specimens. Figure 16 illustrates this notch-strengthening effect by comparing smooth and notch tensile strength for air and consumable electrode vacuum-melted modified H-11 (Potomac A) with notches of varying severity. This plot further illustrates the superior notch tensile strength of consumable electrode vacuum-melted H-11 steel when tested in each direction and with each notch radius regardless of severity. A comparison of notch tensile ratios in Table IV illustrates the consistency of improvement in notch strength realized from consumable electrode vacuum-melting.



Frequency	0-0.1-0.2	0.3-0.4	0.5-0.6	0.7-0.8	0.9-1.0	1.1-1.2	1.3-1.4	1.5-1.6	1.7-1.8
1.1	(a)								3.6 %
1.0									
0.9									0.7%
0.8								1.5%	
0.7									0.7%
0.6						0.7%		0.7%	0.7%
0.5					0.7%	1.5%			
0.4				1.5%	2.2%			0.7%	
0.3			2.2%	2.2%	0.7%	1.5%	0.7%		0.7%
0.2		2.2%	0.7%	0.7%	1.5%				
0-0.1	63.7%	3.6%	1.5%						2.2%

Severity

Frequency	1.0-2.0	3.0-4.0	5.0-6.0	7.0-8.0	9.0-10.0	11.0-12.0	13.0-14.0	15.0-16.0	17.0-18.0
11.0	(b)					2.3%	2.3%		4.7 %
10.0					2.3%	2.3%			
9.0									
8.0									
7.0									
6.0			4.7%						
5.0									
4.0		9.3%							4.7%
3.0									7.0%
2.0	7.0%			2.3%					2.3%
0-1.0	20.9%	11.6%	4.7%	2.3%					9.3%

Severity

FIG. 18.—Comparison of Cleanliness of High-Strength Steels: (*a*) Vacuum Consumable Electrode Processed (Ladish D6AC) and (*b*) Air Melt Processed (Ladish D6A). Note: Severity for Chart (*b*) is 10 times scale on Chart (*a*).

Results of acceptance testing during the period of Jan. 1960, to May, 1960. Bivariate distribution of frequency and severity per unit area.

The per cent improvement in notch strength ratio realized from Consutrode melting is illustrated in Fig. 17 with from 9 to 59 per cent higher notch strength ratio found in H-11 steel melted by this process. This graph also illustrates the relatively greater improvement found in the short transverse direction, for which a comparable value in sheet material cannot now be obtained.

MR. J. P. SHEEHAN[15] (*presented in written form*).—A question arises concerning the effect of carbon content that perhaps the authors might be able to clarify. In discussing the practical significance of the results, they indicated that 300M steel with a nominal carbon content of 0.43 per cent had a notch strength ratio of about 0.38 at a tensile strength level of 275,000 psi. Reducing the carbon content to 0.35 per cent increases the notch strength ratio at 275,000 psi to about 0.7. Fracture toughness in terms of K_c values shows similar trends. Figure 6 of the paper shows that to obtain 275,000 psi tensile strength in a 0.43 per cent carbon 300M steel requires a tempering treatment in the range of 800 to 900 F, whereas the 0.35 per cent carbon 300M steel requires a tempering treatment of around 500 to 600 F to obtain the same strength level. The question arises, therefore, "Is the improvement in notch toughness associated with the lower carbon content steel merely the effect of the carbon content per se or might it not be complicated by the fact that the 0.43 per cent carbon steel is tempered in a tempering range that is known to produce embrittlement whereas the lower carbon content steel is tempered in the recommended tempering temperature range for this particular alloy?"

---

[15] Supervisor, Ferrous Metallurgy, Armour Research Foundation of Illinois Institute of Technology, Chicago, Ill.

MR. C. K. DAVID.[16]—In connection with Mr. Dyrkacz's discussion, the accompanying Fig. 18 shows some data regarding the magnetic particle testing of our Ladish D6A material, both consumable-melted and air-melted. The chart on the right is for air-melted material. This is for some 9 heats and the chart on the left is for consumable material, some 62 heats. The consumable heats are all large ingot heats and it will be noted that the scale on the chart on the right, the air-melt chart, is 10 times that of the consumable-melted. In other words, the whole consumable-melted chart will fit in the bottom left-hand small square of the air-melt chart. This shows that the consumable-melted material in a frequency severity rating type of magnetic particle step down test is considerably cleaner on a statistical basis—based on melting of large ingots.

Would the author care to comment on the relative effect of ingot size on the cleanliness? I noticed in the paper that the ingots used vary from 4 in. 100-lb ingots up to 10-, 12- and, in one case, 24-in. ingots. There is considerable difference between 4-, 10-, 12-, and 15-in. ingots and 26-, 30-, and 32-in. ingots.

MR. E. S. JONES (*by letter*).[17]—Several points in the paper bear additional comments, especially from the viewpoint of those actually engaged in manufacture of high-strength parts from these particular steels. In particular, the comparison of vacuum versus air melting deserves more elaboration.

The authors leave the impression, based on their results, that there is not a great deal of difference in properties between air- and vacuum-melted material and hence there is reason to doubt the need for using vacuum-melted material. To those who have been concerned with the manufacture of high-quality jet engine, aircraft, and rocket parts for the past few years, the merits of vacuum-melted steels are many and proved by experience. Vacuum melting reduces the oxygen, nitrogen, and hydrogen content of the material, improves homogeneity and, though not well documented, removes certain types of undesirable volatile "tramp" elements. The lowered gas content usually results in a much cleaner microstructure, and it is this characteristic that makes its use virtually essential when high strength and high reliability are desired. Occasionally one may encounter "dirty" vacuum-melted heats, but this is uncommon with experienced melters.

The authors stated that both the vacuum- and air-melted steels used in their study were of high cleanliness. This similarity undoubtedly explains to a large degree the similarity in properties found. However, if several heats of air- and vacuum-melted material are carefully examined for microstructural cleanliness as well as smooth and notched tensile properties, the difference in the quality of the two types of melting methods will be readily apparent. Perhaps the best test of all is to request several steel producers to quote on a steel specification which requires a very low inclusion rating. If given the choice between air and vacuum melting, their selection of vacuum melting would be nearly unanimous.

It has been pointed out previously[18] that cleanliness becomes increasingly important as the strength level increases. Microstructural inclusions, stringers, and so on can act as sites of potential stress concentrations which should and can be avoided by vacuum melting. At a yield strength level of about 200,000 psi, 300M is not nearly as sensitive to lack of microstructural cleanliness as at the 230,000 to 240,000 psi yield strength level. Thus, while the microstructural inclusions associated with air-melted material may be tolerable at the lower strength level, it is necessary to achieve a much cleaner microstructure at the higher strength level. Recent Aerojet General Corp. data[19] on several air- versus vacuum-melted steels clearly show the superiority of the latter in notch properties, especially at high strength levels.

---

[16] Metallurgical Engineer, Metallurgy, Ladish Co., Cudahy, Wis.

[17] Manager, Alloys Studies Unit, General Electric Co., Cincinnati, Ohio.

[18] E. S. Jones, "Rocket Materials—A Challenge for Metallurgists," *Journal of Metals*, June, 1960.

With regard to the findings of the authors on carbon content, their data indicate that 300M has a greatly increased sensitivity to sharp notches as carbon content is increased between about 0.30 to 0.45 per cent. While this is probably correct *for the conditions of heat treatment studied*, it must also be recognized that grain size, amount of carbon in solution, and prior microstructural condition all can influence the *level* of notch sensitivity. It has been our experience that changes in austenitizing temperature can have marked effects on the notch to smooth tensile ratio of 300M. While our tests have been on edge notched specimens with a $K_t = 5$ stress concentration, it seems likely that the same differences would be evident, and perhaps would be magnified, at a $K_t = 12$ stress concentration. Associated with rolling and austenitizing temperature is, of course, the grain size of the alloy being studied. It would perhaps be quite revealing if the authors would compare grain sizes on the 300M and H-11 heats and heat treatments used in their study. It seems quite likely that a rather large range of grain sizes probably existed, which could perhaps account for the unexpected behavior of the H-11 steel.

MR. J. A. KIES.[20]—These are fine results but I would like to plead for some additional answers which people do not usually think of in saying whether or not consumable electrode-melting is better. One of these items is the maximum size of the inclusions that one has a right to expect in a completed structure, not just a little specimen. These are the flaws which will generate the initiating cracks at really high stress levels of 240,000 and 300,000 psi. The usual type of ASTM rating for inclusions[21] does not tell us the answer to that question.

Another thing I would like to note is that we need to find out as soon as we can what the improvement in toughness is in the heat-affected zone as well as in the base plate as a result of consumable electrode melting.

We also want to know whether it pays to go to two meltings in vacuum. Is there any improvement to be gained by taking the induction melt and then vacuum melting again in the consumable electrode process?

MESSRS. J. L. SHANNON, G. B. ESPEY, A. J. REPKO, AND W. F. BROWN, JR. (*authors' closure*).—It is extremely gratifying to the authors that so many persons have taken time to study this paper carefully and write stimulating discussions. There is certainly as much interesting information in the discussions as in the paper, and this is as it should be.

The comments of the discussers might be conveniently divided into three categories: (1) those which relate to the general significance of sharp-notch tests and fracture toughness measurements; (2) those dealing with the specific influences of carbon content, and (3) those which consider the effects of vacuum melting. We will consider each category separately as follows.

---

[19] Aerojet General Corp., *Report SRP187*, Nov., 1959.

[20] Physicist, U. S. Naval Research Laboratory, Washington, D. C.

[21] Recommended Practice for Determining the Inclusion Content of Steel (E 45 – 51), 1958 Book of ASTM Standards, Part 3.

General Significance of the Sharp-Notch Test:

The discussions of Drs. Hamaker and Roberts, Prof. Klier, and that of Mr. Pall are mainly concerned with this subject. In part these discussions raise questions which were not intended to be answered by the present investigation and require the introduction of additional concepts. However, these questions are important and we will attempt to reply.

We do not agree with Drs. Hamaker and Roberts that very sharp notch data for all steels are essentially the same function of the strength level within the statistical accuracy of the particular notch test considered. It would seem that such a claim, if true, would throw doubt on all the effects of carbon discussed in this paper. However, the discussers do not dispute these effects.

In our experience, the sharp-edge-notch tensile strength ratio varies about ±10 per cent in a single heat of aircraft quality steel. Larger scatter, of course, will be produced if the structure has a transition temperature at or very near room temperature. However, for the alloys considered here, such conditions correspond to tempering temperatures near 1100 F and therefore rather low strength values. In order to illustrate the difference between H-11 modified and 300M steels at 0.34 per cent carbon level we have shown the ±10 per cent scatter bands of the sharp-edge-notch strength ratio in the accompanying Fig. 19. It would appear to us that there is a distinct difference in toughness between these two steels at strength levels above about 260,000 psi and that this difference increases with the strength level. Regarding the question of heat to heat variations, we have no way of evaluating

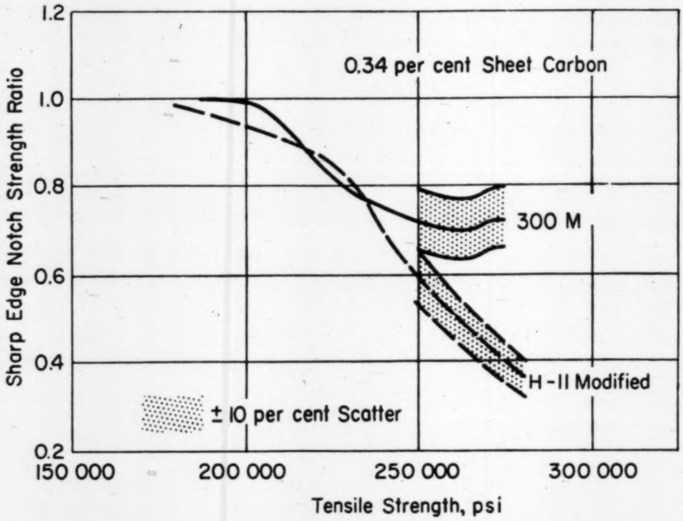


FIG. 19.—Comparison of Sharp-Edge-Notch Tensile Strength Ratios for 300M and H-11 Modified Steels as a Function of Strength Level at 0.34 per cent Carbon Level.

scatter from this cause. Our experience with H-11 modified steel indicates quite uniform behavior in a given carbon range and we have no reason to believe that other steels made with the same care are worse in this respect.

Messrs. Hamaker and Roberts indicate there may be a complete range of notch sharpness values having significance in engineering design. We cannot agree with this philosophy if the influence of cracks is to be considered, and we have discussed this point in detail in the authors' closure to another paper.[22] It is possible that a notch length of only 0.010 in. is critical to the design of certain components. This figure appears to be taken from the experience of Pratt & Whitney Aircraft Co.[23] Apparently they believe that the flaws in their components will not exceed this value, and we have no reason to doubt them. However, we do not see how this in any way detracts from the value of the sharp-notch test; it only means that materials with rather low fracture toughness or notch strength ratio values may be employed if the design and inspection are right.

It is interesting to examine this point further from the standpoint of the required $\mathcal{G}_c$ values ($\mathcal{G}_c = K_c^2/E$). The authors do not know how Hamaker and Roberts proceeded with their calculations. However, we would use the following relations:

$$\mathcal{G}_c = \frac{\pi\sigma^2 a}{E} \quad \ldots\ldots\ldots\ldots\ldots (3)$$

where σ is the average circumferential wall stress calculated from membrane equations, and a is the half length of the crack corrected for local plasticity at the tip. The relation between the observed crack size a_0 and the corrected value a is as follows:

$$a = a_0 + \frac{E\mathcal{G}_c}{2\pi\sigma_y^2} \quad \ldots\ldots\ldots\ldots (4)$$

where σ_y is the yield strength. Now substituting Eq 4 into Eq 3:

$$\mathcal{G}_c = \frac{\sigma^2\pi a_0}{E\left(1 - \dfrac{\sigma^2}{2\sigma_y^2}\right)} \quad \ldots\ldots\ldots (5)$$

If we assume that H-11 modified steel is used at a σ_y of 225,000 psi, that $2a_0 = 0.010$, and that the operating stress equals the yield strength, then the required $\mathcal{G}_c = 53$ in.-lb per sq in. ($K_c = 40 \times 10^3$ psi $\sqrt{\text{in.}}$). According to Fig. 3(*b*) of the present paper, $K_c \sim 80 \times 10^3$ psi $\sqrt{\text{in.}}$ ($\mathcal{G}_c \sim 200$ in.-lb per sq in.[24] can be obtained by tempering 0.40 per cent carbon H-11 modified at 1050 F. The corresponding yield strength is about 225,000 psi. Therefore, on the basis of sharp-notch tests and stress analysis, H-11 modified in 0.063-in. thickness has adequate toughness for the application, providing, of course, the defects are not more than 0.010 in., and do not grow during hydrotest. Now let us examine what might happen if still higher yield strength values were desired from the 0.40 per cent carbon material. According to Hamaker and Vater,[5] 950 F tempering will give 240,000 psi yield strength.[25]

---

[22] G. B. Espey, M. H. Jones, and W. F. Brown, Jr., "Sharp-Edge-Notch Tensile Characteristics of Several High-Strength Titanium Sheet Alloys at Room and Cryogenic Temperatures," Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials, *ASTM STP No. 287*, Am. Soc. Testing Mats., p. 74 (1960).

[23] M. E. Shank, C. E. Spaeth, V. W. Cooke, and J. E. Coyne, "Solid Fuel Rocket Chambers for Operation at 240,000 psi and Over," Rocket Motor Case Symposium, Pratt & Whitney Aircraft Co., Hartford, Conn., Sept. 29–30, 1959.

[24] The thickness of the alloy in the Pratt & Whitney investigation was 0.070 in. for full-scale vessels, and to be strictly correct the K_c values should be obtained for this thickness. However, this does not upset the general argument.

[25] We were never able to exceed about 235,000 psi; however, small differences in residual stresses and testing technique might account for this lower value.

Now using Eq 5, the required $\mathcal{G}_c$ = 60 in.-lb per sq in. ($K_c = 43 \times 10^3$ psi $\sqrt{\text{in.}}$). According to Fig. 3(*b*) this fracture toughness is at best very marginal for H-11 modified steel tempered at 950 F. On the other hand, if we consider 300M at the 0.40 per cent carbon level according to Fig. 4 of the paper, 240,000 psi yield is available at a tempering temperature of 600 F and according to Fig. 3(*a*) the corresponding $K_c = 115 \times 10^3$ psi $\sqrt{\text{in.}}$ ($\mathcal{G}_c$ = 440 in.-lb per sq in.), which gives a considerable safety factor. In fact, this material could withstand a crack about 0.070 in., seven times as long as the maximum size specified. It is to be emphasized that none of these calculations could be made from data on mildly notched specimens by any method known to us.

We do not feel unduly alarmed by the results from sharp notch tests. In the example given above they show 0.063-in. H-11 modified steel to be adequate at 225,000 psi yield strength level for maximum defect sizes of 0.01 in. However, quoting from the Pratt & Whitney Symposium paper by Shank et al:[23] "Finally a word must be said about design philosophy . . . with critical crack lengths of 0.010 inch no detail is unimportant . . . one must employ such practices as making bosses and skirts integral with the main members of the missile casing and eliminating longitudinal weld seams. Admittedly these measures are expensive; they do, however, insure integrity in service." Under such conditions it is only logical to employ the toughest materials available, if they are satisfactory in other respects.

We are not quite sure we understand completely the comments of Mr. Pall. He apparently feels that $\beta \geq 8$ is required for satisfactory pressure vessel performance rather than $\beta \geq 2\pi$ as suggested by Irwin (reference (2) of the paper). We agree that a wider specimen would be required to yield meaningful K_c values under such conditions. Mr. Pall then presents arguments to show that the data in Fig. 3(*a*) for 300M (0.40 per cent carbon) yields a β value of 3.78, which is inconsistent with a relation between per cent shear and β which was suggested by Irwin. This relation was established on the basis of data covering only a few steel types. On the basis of the information at hand, it appeared that steels exhibiting above about 80 per cent shear should have β values at least equal to 2π. The difficulty with 0.40 per cent carbon 300M is that the specimens exhibit 100 per cent shear but have β values less than 4. We doubt whether a useful relation exists between β and the per cent shear and this has been discussed by one of the authors elsewhere.[26] Furthermore, it is not clear to the authors that any arbitrary limiting value can be set on β for successful pressure vessel performance. For example, consider the H-11 modified steel used in the above discussed Pratt & Whitney pressure vessels. In the tempered condition used, $K_c \sim 80 \times 10^3$ psi $\sqrt{\text{in.}}$ and $\beta \sim 2.0$ for 0.063-in. stock. This is certainly well below any limiting value proposed so far and the shear is less than 30 per cent. The problem here is that we really do not know how embedded cracks grow.

Mr. Klier raises two questions. With regard to the first, we are not certain what he means by "unambiguous" measurement of K_c. One limitation to the measurement of a meaningful value is that the net fracture stress (maximum load divided by the area remaining at the start of unstable fracture) shall be less than the yield strength. When this condition is not met the values might be called ambiguous. We did not report such values. The maximum K_c which can be measured depends on the specimen width and the yield strength. Its approximation is:

---

[26] W. F. Brown, Jr., "Mechanical Screening Tests for Sheet Alloys," *Proceedings*, Seventh Sagamore Ordnance Materials Research Conference, Syracuse University, Syracuse, N. Y., Aug., 1960.

$$K_{c\,\mathrm{max}}^2 = 0.38\sigma_y^2 W$$

where σ_y is the yield strength and W is the specimen width. To evaluate the present materials in their low yield strength but tougher conditions we could have increased the specimen width. Dr. Klier's second question pertaining to the interchangeability of K_c measurements cannot be answered without further tests on various types of specimens. The authors are cooperating with members of the Naval Research Laboratory on just such a program for sheet materials.

Influence of Carbon Content:

The authors are pleased that Mr. Carman's results confirm their observations regarding the effect of carbon content on the toughness of silicon modified low-alloy steels. Mr. Carman's data demonstrate that the superior toughness of the lower carbon material is maintained at subzero temperatures.

Both Mr. Sheehan and Mr. Jones raise the point that the reported effects of carbon could be clouded by other factors. Regarding Mr. Sheehan's question concerning the effect of tempering 0.43 carbon 300M steel in an "unfavorable range," we would refer to Fig. 4 of the paper. It will be noted that 250,000-psi tensile strength level is obtained for *both* 0.34 and 0.40 per cent carbon 300M by tempering in the "unfavorable" range between 800 and 850 F. Now, referring to Fig. 6 of the paper, substantial improvements in notch sensitivity at 250,000 psi strength level are obtained by reducing the carbon from 0.40 to 0.34 per cent. Thus, while shifts in tempering temperature are probably in part responsible for the effects observed, the influence of carbon certainly exists. Furthermore, we do not dispute the statement that many factors other than carbon content can influence the notch properties. We did not investigate these other factors, but do not believe that they could in themselves explain away the well-documented effect of carbon.

Messrs. Hamaker and Roberts further state that the high alloy content of H-11 modified steel will permit increase in the carbon content necessary to achieve tensile strength levels above 300,000 psi, with reduction in the notch sensitivity. This conclusion is not entirely substantiated by the data presented in this paper. According to Fig. 7 the beneficial effect of increasing carbon fades out rapidly at strength levels over about 280,000 psi. Furthermore, it was pointed out that if various carbon levels of H-11 modified steel are heat treated to their highest strength level the resulting notch sensitivity is nearly identical and high.

Effects of Vacuum Melting:

All of the discussion on this subject has been in defense of the consumable electrode vacuum process and the discussers apparently feel we have indeed done it an injustice. We certainly did not intend to do so. The experiments were carefully designed to answer the question as to whether or not consumable electrode vacuum remelted material in sheet form had better sharp-notch properties than aircraft quality air melt. The data obtained by the authors did not reveal any large improvements in sheet fracture toughness associated with vacuum melting.[27] The mechanical property data presented by the discussers either support this conclusion or are not directly applicable.

The 300M steel sheet data presented by Mr. Dyrkacz for mildly notched specmens show improvement due to consumable electrode vacuum melting to be rather small. A portion or all of this improvement may be associated with the "abnormally" low smooth strength observed for the vacuum-melted alloy. This could have resulted from sheet decarburization which would increase the toughness independent of the melting procedure.

The data presented by Messrs. Borneman and McCaffrey should be considered in light of the following:

1. Their cylindrical specimens represented fracturing under conditions of plane strain and it is not expected that such tests would necessarily give the same results as those obtained with sheet where both the stress state and fabrication history are different.
2. The largest improvements are noted for tests employing a 0.010-in. notch radius. These are interesting and indicate improved service performance in the presence of mild stress concentrations, such as threads, fillets, and so on.
3. Their data for 0.001-in. radius notches best simulate the action of cracks (our major interest in this investigation), and with the exception of the short transverse direction the improvement in notch strength ratio is only about 10 per cent (see Fig. 17 of the discussion). This is a value close to that reported by the authors for H-11 modified sheet at 260,000-psi strength level.

Messrs. Hamaker and Roberts' comment concerning the improvement in transverse ductility of heavy sections brought about by vacuum melting is not disputed in any way by the authors.

Mr. Jones implies that all those concerned with the manufacture of high-quality military hardware believe the merits of vacuum-melted steels are many and proved by experience. Such a very broad statement hardly supports the argument. The Aerojet data[19] are contained in a company report not available to us. The data in the paper by Jones[18] were obtained using specimens having mild notches ($K_t = 5$) and are not necessarily indicative of fracture toughness.

The remainder of the discussion on vacuum melting is primarily concerned with steel cleanliness. In this connection the authors appreciate the information presented by Messrs. Dyrkacz and David which shows that consistently higher cleanliness can be obtained by the consumable electrode vacuum melting process. The authors did not make inclusions studies. These would have been in order if significant differences were found between air- and vacuum-melted material. Unfortunately, at best such an investigation in sheet is extremely difficult. Our experience indicates the inclusion content varies with specimen position and from sheet to sheet of the same heat. The problem is therefore of a statistical nature and requires a very large number of tests.

There is no reason for us to dispute these claims for higher cleanliness in vacuum-melted alloys; however, there is no conclusive proof that this results in higher fracture toughness in sheet material. The proof for this will have to await the time when sharp-notch tests are incorporated into quality control procedures.

Perhaps some of the difficulty in the argument over the merit of vacuum melting lies in the lack of a clear understanding of the sharp-notch test and the

---

[27] This observation applies equally well to either the transverse or longitudinal direction. Both longitudinal and transverse data were obtained for all vacuum-melted material (see Figs. 2 and 3 of the paper) and enough such data were obtained for air-melted sheet to indicate negligible directionality existed for either melting practice.

concept of fracture toughness as defined by K_c. Increasing the cleanliness certainly reduces the probability of encountering an inclusion of sufficient size that would in itself cause unstable fracturing. The possibility of such an occurrence would of course increase with the strength level or with any other factor which reduces fracture toughness. On the other hand, the fracture toughness per se may not be appreciably increased by this melting process. This would mean that the strength of a structure in the presence of a crack might be increased providing the crack was small in comparison with the inclusions present. Thus, one might expect improvement in fatigue strength since the major defects frequently are inclusions. Also, improvement might be expected in the strength of heavy sections tested in the short transverse direction where inclusions are unfavorably oriented with respect to the stress and constitute the major flaws present. On the other hand, a rocket motor case containing through the thickness cracks (fabrication defects) of an extent appreciably larger than any inclusion present would be strength limited by the crack alone. Vacuum melting in this case may result in little or no difference in performance.

In conclusion, we agree with the remarks made by Mr. Kies and wish to add that a study of the influence of size and shape of inclusions will be necessary before we can successfully correlate cleanliness data with strength properties.

MECHANICAL AND PHYSICAL PROPERTIES OF FIVE COPPER-BASE CASTING ALLOYS*

BY W. H. JOHNSON[1] AND J. G. KURA[2]

SYNOPSIS

Accurate measurements were made for the mechanical and physical properties of 76 Cu-2½Sn-6½Pb-15Zn alloy, 20 per cent nickel silver, 81-4-15 silicon brass, 65,000 psi manganese bronze, and 110,000 psi manganese bronze alloys at various temperatures ranging from −40 to 550 F. The properties measured were ultimate strength, yield strength, elongation, reduction of area, modulus of elasticity, compressive strength, V-notch Charpy impact strength, Brinell hardness, fatigue, machinability, melting range, patternmaker's shrinkage, density and specific gravity, electrical resistivity, thermal conductivity, and thermal expansion.

This paper constitutes the second portion of a continuing program to provide accurate measurements for mechanical and physical properties of sound test bars from good-quality melts of commercial alloys. Data reported earlier (**1**)[3] were for a group of three casting alloys which were identified as 2A, 3A, and 4A by the ASTM Specification B 30 - 59[4] for ingots, and the trade designations were Navy "M," 80-10-10, and 85-5-5-5, respectively.

Data on a second group comprising five alloys are reported in the present paper. The five alloys are identified as 5B, 8A, 8C, 11A, and 13B by the ASTM Specification B 30 – 59.[4] The casting specifications are ASTM B 145 – 52, alloy 5B[5]; B 147 – 52, alloy 8A and 8C[6]; B 149 – 52, alloy 11A[7]; and B 198 – 58, alloy 13B,[8] respectively. These alloys are referred to in this report as 76Cu-2½Sn-6½Pb-15Zn, 65,000 psi manganese bronze, 110,000 psi manganese bronze, 20 per cent nickel silver, and 81-4-15 silicon brass, respectively. In presenting the data, the alloys are considered in the following order:

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Senior Metallurgist, Nonferrous Metallurgy Division, Battelle Memorial Inst., Columbus, Ohio.

[2] Chief, Nonferrous Metallurgy Division, Battelle Memorial Inst., Columbus, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

[4] Specification for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 59), 1959 Supplement to Book of ASTM Standards, Part 2, p. 1.

[5] Specification for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 52), 1958 Book of ASTM Standards, Part 2 p. 245.

[6] Specification for High-Strength Yellow Brass (Manganese Bronze) and Leaded High-Strength Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 – 52), 1958 Book of ASTM Standards, Part 2, p. 253.

[7] Specification for Leaded Nickel Brass (Leaded Nickel Silver) and Leaded Nickel Bronze (Leaded Nickel Silver) Sand Castings (B 149 – 52), 1958 Book of ASTM Standards, Part 2, p. 263.

[8] Specification for Silicon Bronze and Silicon Brass Sand Castings (B 198 – 58), 1958 Book of ASTM Standards, Part 2, p. 344.

(1) 76 Cu-2½ Sn-6½ Pb-15 Zn alloy
(2) 20 per cent nickel silver,
(3) 81-4-15 silicon brass,
(4) 65,000 psi manganese bronze, and
(5) 110,000 psi manganese bronze

It should be pointed out that alloys with many variations in composition may meet a given ASTM specification for chemical composition. The variations in composition could result in marked changes in mechanical properties. For example, in the 81-4-15 silicon brass, an alloy with a silicon content on the low end of the range of the specification will have higher ductility. In another example, a 65,000 psi manganese bronze with a copper content on the low end of the range of the specification, or aluminum on the high end of the range, or both, will have higher tensile strength. Therefore, the specific composition of the five types of alloys used in the investigation were intended to be representative of the ones that are used widely by the industry. In spite of this intention, a manganese bronze of the 8A class was initially studied which met the requirements of chemical composition for the ASTM specification, but the tensile properties were much higher than would normally be expected by the industry for this type of alloy. New ingot, identified as lot B, of slightly different composition from the previous ingot, lot A, was evaluated to provide data representative of the type of alloy that is used commercially.[9] Only those properties that were expected to be affected by the slight change in chemical composition between the lots of ingot were re-evaluated. In each instance, the data for a specific property are identified with the ingot lot that was used.

Of the 16 mechanical and physical properties that were determined, some were measured only at room temperature, but most of them were measured at −40 and 75 F, and at two elevated temperatures up to 550 F. Except where specifically noted otherwise, the methods that were used to measure the properties were those that were described in the previous paper (1). Determination of the creep properties was part of the program, but the results will probably be presented in a later paper.

EXPERIMENTAL PROCEDURE

Preparation of Specimen:

Specimens for determining the patternmaker's shrinkage were cast in baked sand molds. All of the other specimens were cast in green sand molds.

The following casting designs provided specimens for evaluation of the different properties:

1. Double-horizontal full-web type (ASTM Recommended Practice B 208 − 58[10], Fig. 3).
2. Modified 1-in. web-Webbert (1).
3. Double keel block.[10]
4. Patternmaker's shrinkage bar (2).

The double-horizontal full-web test bar was used for the low-shrinkage alloys: 76-2½-6½-15, 20 per cent nickel silver, and the 81-4-15 silicon brass. The modified 1-in. web-Webbert test bar was used when the size of the desired test specimen from these three alloys was too large to be machined from the double-horizontal full-web type. The double horizontal full-web test bars were used to determine the melt quality, tensile properties, hardness, melting range, and density. All of the other properties except patternmaker's shrinkage were measured from the modified 1-in. web-Webbert test bars.

---

[9] Chemical composition of the two lots is given in Table II.

[10] Recommended Practice for Tension Test Specimens for Copper-base Alloys for Sand Castings (B 208 − 58), 1958 Book of ASTM Standards, Part 3, p. 98.

Patternmaker's shrinkage was determined with the special casting procedure developed by Pilling and Kihlgren (2).

The keel block casting was used for the two manganese bronzes which are considered to be high-shrinkage alloys.

TABLE I.—EFFECT OF CASTING DESIGN ON ROOM-TEMPERATURE TENSILE PROPERTIES.

Design	Pouring Temperature, deg Fahr	Tensile Strength, psi			Elongation, per cent		
		Max	Min	Avg^{a,b}	Max	Min	Avg^{a,b}
76-2½-6½-15							
Double-horizontal full web	1855	36 600	36 100	36 400^{4}	38	30	34^{4}
Modified 1-in. web-Webbert	1855	35 200	30 100	33 100^{4}	30	24	28^{4}
20 PER CENT NICKEL SILVER ALLOY							
Double-horizontal full web	2350	46 700	43 700	45 200^{6}	20	15	17^{6}
Modified 1-in. web-Webbert	2335	46 600	43 700	45 200^{6}	20	16	18^{6}
81-4-15 SILICON BRASS ALLOY							
Double-horizontal full web	1770	72 200	68 500	69 800^{6}	21	17	19^{6}
Modified 1-in. web-Webbert	1770	71 500	67 900	69 700^{5}	23	19	21^{5}
65 000 PSI MANGANESE BRONZE							
Double-horizontal full web	1850	71 900	71 500	71 700^{6}	39	35	37^{6}
Keel block	1850	71 500	71 500	71 500^{6}	38	35	37^{6}
110 000 PSI MANGANESE BRONZE							
Double-horizontal full web	1940	120 500	120 500	120 500^{2}	17	16	17^{2}
Keel block	1930	120 000	119 500	119 800^{2}	20	16	18^{2}

[a] The standard deviation for tensile strength is ±285 psi; that for elongation, ±½ per cent. (See under "Reliability of Data" for method used to calculate the standard deviation.)

[b] Superscript numbers give the number of specimens.

For the three low-shrinkage alloys, it was necessary to determine whether the tensile properties of the modified 1-in. bar were comparable with those of the double-horizontal bar. Therefore, both types were cast from the same melt. The pouring temperatures were in a predetermined range which is described later. The data in Table I show that only in the case of the 76-2½-6½-15 alloy were the tensile properties somewhat lower for the modified bar than for the double-horizontal bar. Similar experiments comparing the keel block and the double-horizontal bar when cast from the low-strength and high-strength manganese bronzes (Table I) showed that the two test-bar casting designs gave practically identical values. It was concluded that the modified 1-in. bars were acceptable for measuring the properties of the three low-shrinkage alloys, and the double-horizontal bars were acceptable for evaluating the melt quality of all five of the alloys.

Melting and Selection of Pouring Temperature:

The first step was to determine the pouring temperature at which the optimum tensile properties were obtained in the castings of each of the alloys. Initially, the melts were prepared in a silica-lined, high-frequency induction furnace and were transferred to a ladle for pouring. During melting, a thin cover of glass was used only on the nickel silver alloy to prevent excessive zinc loss by oxidation while the melt was being superheated. Double-horizontal castings were poured from each melt of the three low-shrinkage alloys at four or more temperatures. Keel block castings were poured at five different temperatures from the melts of the two types of manganese bronze alloys.

Later experiments revealed that the 76-2½-6½-15 and the nickel silver alloy had to be prepared in a gas-fired furnace to obtain consistently high melt quality. A clay-graphite crucible without a cover over the melt and a slightly oxidizing atmosphere were used. The pouring temperature range in which the best combination of tensile strength and elongation was obtained for each of the five alloys was as follows:

Alloy	Pouring Temperature Range, deg Fahr
76-2½-6½-15[a]	1900 to 1850
20 per cent nickel silver[a]	2400 to 2375
81-4-15 silicon brass[b]	1800 to 1750
65 000 psi manganese bronze[b]	1875 to 1825
110 000 psi manganese bronze[b]	1950 to 1900

[a] Prepared in a gas-fired furnace.

[b] Prepared in a high-frequency induction furnace.

The ingot melting stock for each alloy met the prescribed ASTM specification for composition.[4] The number of castings poured from each melt was limited to six or seven, so that all castings could be poured within the pre-established temperature range. The shake-out time for all castings throughout the program was ½ hr.

The average analyses for the compositions of the various melts in the program are presented for each type of alloy in Table II. All of the melts were within the composition range specified in the ASTM specifications.[5, 6, 7, 8]

The results of tests on melt quality conducted on specimens machined from the double-horizontal full-web test-bar castings are summarized in Table III. In all cases, the tensile properties of the castings exceeded the minimum set by ASTM specifications.[5, 6, 7, 8]

In addition to determining the composition and quality of each melt, specimens were radiographed for internal defects before they were subjected to long-time tests such as fatigue and creep.

Reliability of Data:

All of the individual values for a specific property for each alloy were treated simultaneously in a statistical manner (3). This gave the standard deviation of $\pm 1\ \sigma$ for each average value listed in the footnotes of the various tables.

DATA ON PROPERTIES

Tensile Properties:

The tensile properties of the five alloys were measured on bars in duplicate at −40 F, room temperature, and at two elevated temperatures. The tension specimen conformed in detail to Fig. 5 of ASTM Recommended Practice B 208 - 58.[10]

Hydraulic testing machines were used which are accurate to ±0.5 per cent. The rate of straining was accurately controlled at 0.003 in. per in. per min to the yield strength. Previously, (1), the rate of straining was estimated to be 0.002 in. per in. per min. Above the yield strength, the crosshead speed was about 0.05 in. per min. The specimens were held at the testing temperature to within a maximum of ±5 F for at least 20 min prior to load application. However, the variation in temperature was usually only about ±2 F.

Strain on the tensile specimen at all temperatures was determined by means of an extension-type microformer extensometer which was attached to the specimen over the 2-in. gage length. The sensitivity of the extensometer was 0.000004 in. per in. of gage length. An autographic recorder plotted the load-strain curve. Compared with the previous program (1), the improved techniques used generally decreased the standard deviation in the experimental data.

Data on the tensile properties are presented in Fig. 1. The ultimate strength of all of the alloys generally decreased steadily with increasing temperature. The exception was the silicon brass which retained its strength up to 450 F, but decreased rapidly thereafter. Of the five alloys the 110,000 psi manganese bronze was the only one which revealed a wide difference in yield strength

TABLE II.—AVERAGE CHEMICAL ANALYSIS FOR CASTINGS FROM EACH ALLOY TYPE.[a]

Element	Chemical Analysis, per cent					
				Manganese Bronze		
	76-2½-6½-15	20 per cent Nickel Silver	81-4-15 Silicon Brass	65 000 psi		110 000 psi
				Lot A	Lot B	
Copper	75.8	63.8	80.7	57.84	59.6	61.6
Tin	2.7	3.8	<0.01	0.26	0.27	...
Lead	5.6	4.2	<0.01	0.17	0.14	...
Zinc	14.8	7.1	15.1	39.3	37.3	25.9
Iron	0.26	0.74	0.10	1.0	1.5	2.8
Antimony	0.13	0.10	...	...	...	...
Nickel	0.66	20.2	<0.01	0.08	...	...
Aluminum	...	...	...	1.07	1.1	5.9
Manganese	...	0.03	0.01	0.5	0.4	3.8
Silicon	...	...	4.1	...	...	...
Sulfur	0.05	...	...	...	...	...

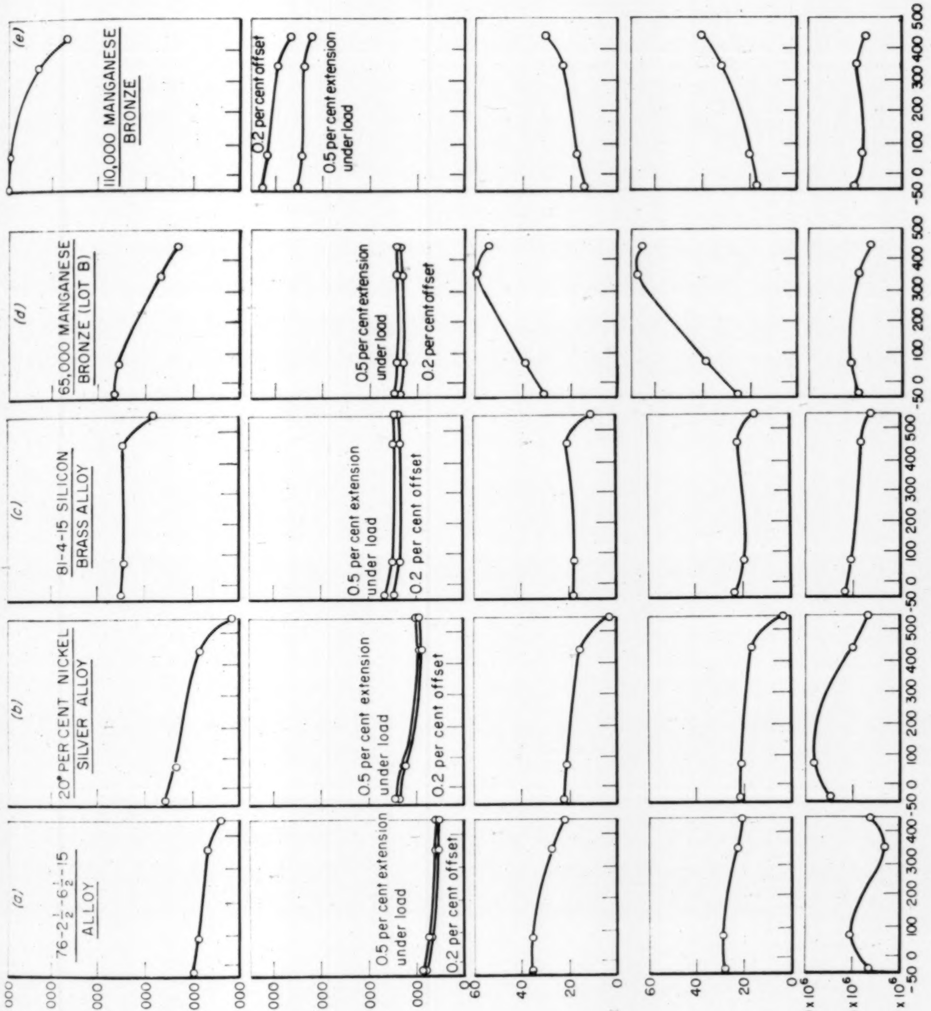
[a] Fourteen heats each of 76-2½-6½-15 and nickel silver alloys, seventeen of silicon brass, eight of each lot of 65,000 psi manganese bronze, and twelve of 110,000 psi manganese bronze.

TABLE III.—SUMMARY OF THE ROOM-TEMPERATURE TENSILE PROPERTIES DETERMINED FOR MELT-QUALITY EVALUATION OF THE FIVE ALLOYS.

Alloy	Tensile Strength, psi			Elongation 2 in., per cent		
	Max	Min	Avg[a,b]	Max	Min	Avg[a,b]
76-2½-6½-15	37 800	35 600	36 900[25]	38	30	33[25]
20 per cent nickel silver	48 600	43 800	46 700[28]	23	17	19[28]
81-4-15 silicon brass	72 000	65 400	68 600[28]	23	16	19[28]
65 000 psi manganese bronze (lot B)	72 000	71 000	71 800[14]	38	35	37[14]
110 000 psi manganese bronze	120 500	118 000	119 600[22]	19	15	16[22]

[a] Standard deviation for tensile strength is ±285 psi; that for elongation, ±½ per cent.

[b] Superscript numerals give the number of specimens.



(a), (b), (c) three low-shrinkage alloys.
(d), (e) two high-shrinkage alloys.

FIG. 1.—Tensile Properties of Five Copper-base Casting Alloys.

for the two methods of measuring this property. It was also the exception in that the yield strength was higher by the offset method than by the extension-under-load method. The yield strength for all the alloys appeared to be less sensitive to increase in temperature than the ultimate strength. The ductility of the 76–2½–6½–15 and nickel silver alloy decreased with increasing temperature, but appeared to be stable for the silicon brass up to a temperature of 450 F as shown in Fig. 1(*a*), (*b*), and (*c*). In contrast, Fig. 1(*d*) and (*e*) shows that the ductility of both of the manganese bronze alloys increased with increasing temperature. The modulus of elasticity was somewhat erratic with increase in temperature for the 76–2½–6½–15 alloy and the 110,000 psi manganese bronze. However, the general trend for all of the alloys was a decrease in modulus as the temperature was raised above room temperature.

The accuracy of the individual values is estimated to be as follows:

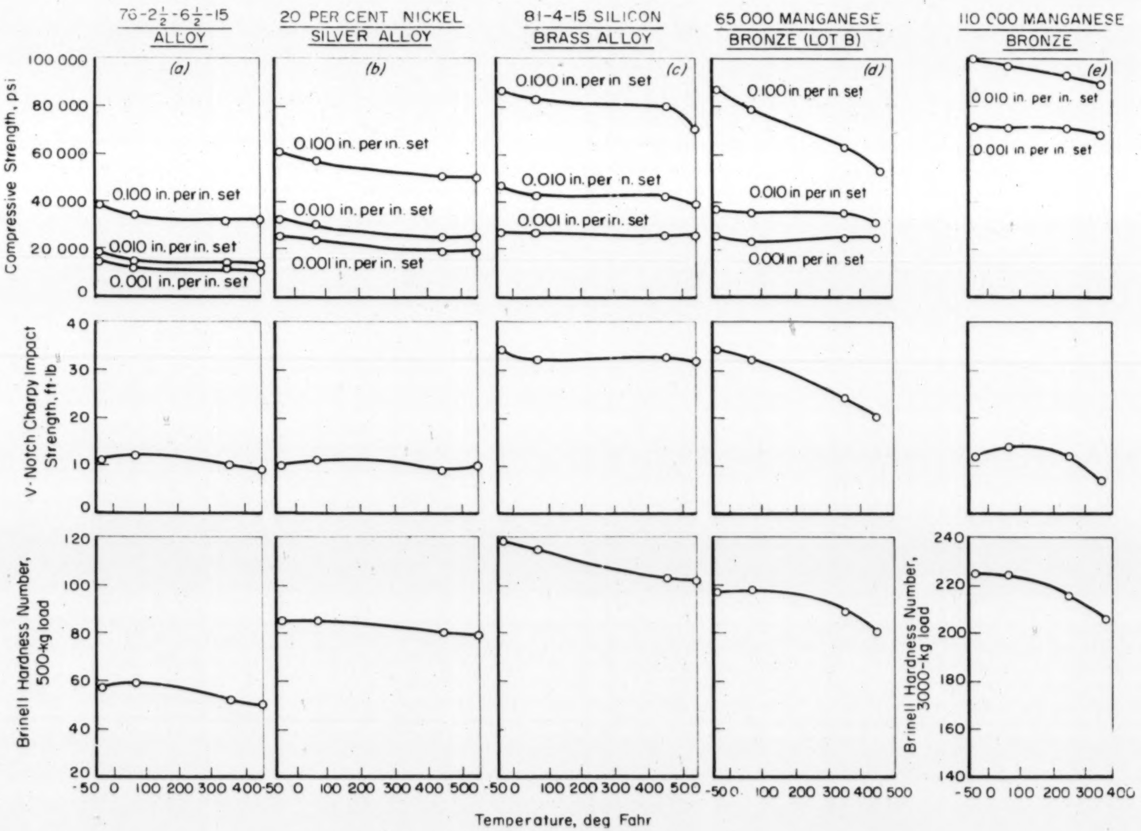
It was stated earlier that the shake-out time for all castings was ½ hr. To show that shake-out time is important with respect to mechanical properties in some alloys, one nickel silver casting was left in the mold overnight. The room temperature properties of this casting are compared below with a casting having normal shake-out time.

Shake-Out Time, hr	Yield Strength, psi		Ultimate Strength, psi	Elongation in 2 in., per cent	Reduction of Area, per cent
	0.2. per cent Offset	0.5 per cent Extension			
18	28 300	30 000	52 800	16	19
½	24 600	26 000	46 900	21	21

It is apparent that the yield strength and ultimate strength were appreciably higher and the ductility was somewhat lower in the slowly cooled casting. In commercial practice, castings of alloys of this type should not be left to cool in the mold for an excessively long period of time, if elongation is a prime consideration.

Property	Temperature, deg Fahr	Experimental Error	Standard Deviation
Ultimate strength[a]	−49 to 550	±1 per cent of the ultimate strength	±285 psi at 75 F, ±600 psi at other temperatures
Yield strength	−40 to 75	±1 per cent of the yield strength	±365 psi
	250 to 550	±2 per cent of the yield strength	±365 psi
Reduction of area	−40 to 550	±1½ per cent	±1 to ⅓ per cent
Elongation[a]	−40 to 75	±1 per cent	±½ per cent at 75 F, ±⅓ per cent at other temperatures
Young's modulus	−40 to 75	±2 per cent of the modulus	±208 560 psi
	250 to 550	±4 per cent of the modulus	±208 560 psi

[a] Twenty-seven specimens were tested at room temperature for the 76-2½-6½-15 alloy, 30 for the nickel silver, 30 for the silicon brass, 16 for the 65 000 psi manganese bronze, and 24 for the 110 000 psi manganese bronze. Specimens in duplicate were tested at all other temperatures and for the other properties listed.



(*a*), (*b*), (*c*) three low-shrinkage alloys. (*d*), (*e*) two high-shrinkage alloys.

FIG. 2.—Compressive Strength, Charpy Impact Strength, and Hardness of Five Copper-base Casting Alloys.

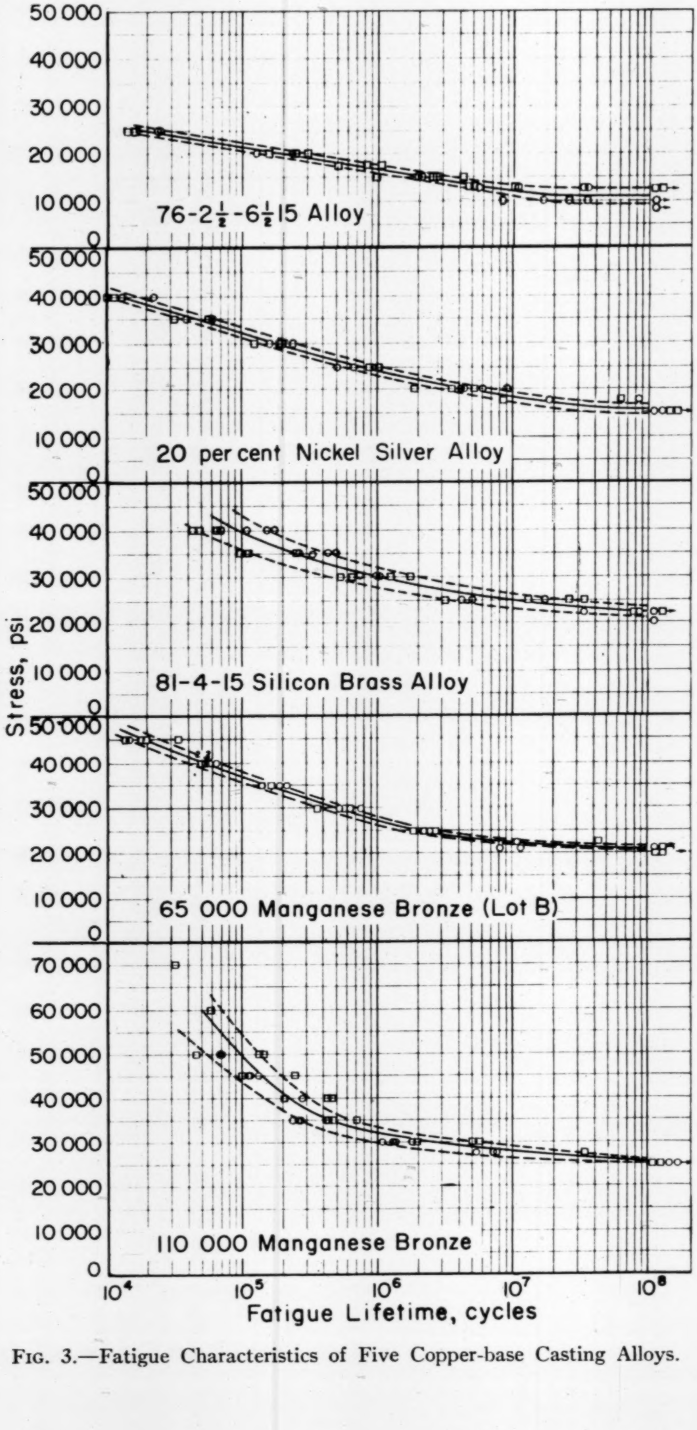


FIG. 3.—Fatigue Characteristics of Five Copper-base Casting Alloys.

Compressive Strength:

Cylindrical specimens $\frac{3}{4}$ in. in diameter and 2 in. long were used. They were machined to ±0.005 in. on the diameter and ±0.005 in. on the length, and the opposite ends were ground to make them parallel to within ±0.0002 in.

The rate of straining with the hydraulic testing machine was 0.001 in. per in. per min below the 0.1 per cent offset yield strength. Above this point the crosshead speed was 0.02 in. per min. The compressive strength was measured from load-strain diagrams at 0.1, 1.0, and 10 per cent offset, which corresponds to permanent sets of 0.001, 0.010, and 0.100 in., respectively. Sensitivity of the extensometer was 0.00003 in. per in. of gage length. The specimens were held at the testing temperature to within ±5 F for at least 20 min.

An accurate measurement of the compressive strength could not be obtained at a permanent set of 0.100 in. per in. for the 110,000 psi manganese bronze alloy. The high load that was required caused the top end of the specimen to slip sideways when the permanent set reached a value of about 0.03 in. per in.

The data in Fig. 2 show that at the greatest permanent set there was a progressive decrease in the compressive strength of all of the alloys as the temperature was increased. The loss in strength was quite marked in the 65,000 psi manganese bronze. However, at the lowest permanent set, very little loss in strength occurred in the five alloys when the temperature was increased above room temperature.

The accuracy of these data is estimated to be as follows:

Compressive Strength[a]	Experimental Error, per cent	Standard Deviation, psi
At 0.001 in. per in. permanent set	±1	±726
At 0.010 in. per in. permanent set	±1	
At 0.100 in. per in. permanent set	±2	

[a] Specimens in duplicate were tested at each temperature level.

Impact Strength:

Standard V-notch Charpy specimens were tested in triplicate. The design conformed to Fig. 3 in ASTM Methods E 23 - 56 T.[11] At the moment of impact, the pendulum was moving at a velocity of 12.75 ft per sec and had an energy of 110 ft-lb.

As shown in Fig. 2 the impact strength of the alloys was relatively insensitive to change in temperature, except for the two manganese bronzes. The impact strength of the 65,000 psi manganese bronze decreased continuously and appreciably as the temperature was increased. The 110,000 psi manganese bronze showed an appreciable drop when the temperature exceeded 250 F. Outstandingly high strength was exhibited by the silicon brass alloy at temperatures up to 550 F.

Hardness:

Brinell hardness measurements were made with a 10-mm diameter tungsten carbide ball on flat surfaces machined on the side of the grip ends of bars that had been used to determine room-temperature tensile properties. Three impressions were made on each of two specimens of each alloy after the specimens were placed in the temperature environment for 30 min.

---

[11] Tentative Methods for Notched Bar Impact Testing of Metallic Materials (E 23 - 56 T), 1958 Book of ASTM Standards, Part 3, p. 69.

Figure 2 shows that a small decrease in hardness occurs gradually for each alloy as the temperature is increased above room temperature. The accuracy of the individual measurements is estimated to be ±1 per cent, and the standard deviation of the average values is ±1 Brinell hardness number.

Fatigue Strength:

R. R. Moore rotating-beam machines were used. The specimens had a minimum section measuring 0.280 in. in diameter, (1). After polishing, the average surface finish was 5 microinches rms. The specimens were loaded as a simple beam while rotating at a speed of 10,000 rpm.

TABLE IV.—MACHINABILITY RATINGS.

Alloy	Machinability Rating Index
Free-cutting brass[a]	100
76-2½-6½-15	178
20 per cent nickel silver	70
81-4-15 silicon brass	50
65 000 psi manganese bronze, lot B	26
110 000 psi manganese bronze	8

[a] Nominal composition of the wrought bar stock was 61.5 per cent copper, 35.5 zinc, 3 lead. See ASTM Standard Specification for Free-Cutting Brass Rod, Bar, and Shapes for Use in Screw Machines (B 16 – 58), 1958 Book of ASTM Standards, Part 2.

The solid curves in Fig. 3 represent the mean value. The broken-line curves represent a standard deviation of ±1 σ. For the five alloys at 100 million cycles, the mean fatigue limit was estimated to be as follows:

Alloy	Mean Fatigue Limit, psi
76-2½-6½-15	11 000
20 per cent nickel silver	15 500
81-4-15 silicon brass	22 000
65 000 psi manganese bronze	21 250
110 000 psi manganese bronze	25 000

Machinability:

Machinability was determined on a lathe in which a constant thrust is applied to the tool carriage by a weight and pulley system, (1). The test bars measured ⅞ in. in diameter by 7 in. in length. Wrought bar stock of free-cutting brass was used as a standard for comparison purposes.

In the previous program (1), machinability ratings were based on tests conducted in quadruplicate from one heat of each alloy. In the present program, the machinability ratings were also based on tests from one heat of each alloy. Even though five tests were made on each alloy, the ratings for the 76–2½–6½–15 alloy and the 110,000 psi manganese bronze alloy diverged widely from the free-cutting brass.

Ideally, numerous samples from many heats should be used to arrive at a truly reliable machinability index rating. Even then the machinability of a specific heat can deviate considerably from the mean rating for an alloy. Therefore, the 76–2½–6½–15 alloy and the 110,000 psi manganese bronze alloy were further evaluated by conducting at least 7 additional tests on bars from a second heat of each alloy.

The machinability ratings are presented in Table IV. The 76–2½–6½–15 alloy had the best machinability. Although the 110,000 psi manganese bronze had quite a low rating index, it does not mean that it is unmachinable; the low rating merely means it is much less machinable than the standard reference alloy. The accuracy of the machinability test is estimated to be ±20 per cent with respect to the standard reference alloy.

Melting Range:

The differential thermal analysis technique was used (1). A copper rod served as the neutral body except for the determination of the melting range for the nickel silver alloy. Because of the high melting temperature of this alloy, a neutral body of molybdenum was used. The alloy test specimen as well as the neutral body were ½ in. in diameter and ¾ in. long.

The data for the melting ranges are presented in Table V and are estimated to be accurate to within ±3 F for all the alloys except the 65,000 psi manganese bronze. The accuracy for this latter alloy is ±5 F. Previous work by Gould and Ray (4) showed that, for the 81–4–15 silicon brass alloy, the beginning and end of melting were 1490 and 1598 F, respectively. The data were obtained by cooling the melt. Their values are lower than those reported in Table V. However, the data in Table V were obtained by heating the specimens and are regarded as a more reliable indication of the liquidus and solidus temperatures of the alloys than data that would be obtained by cooling the melts.

TABLE V.—MELTING-POINT DATA.

Alloy	Beginning of Melting, deg Fahr						End of Melting, deg Fahr		
	Lead Phase			Copper Phase					
	Max	Min	Avg[a,b]	Max	Min	Avg[a,b]	Max	Min	Avg[a,b]
76-2½-6½-15	610	605	607^3	1530	1520	1527^3	1750	1745	1748^3
20 per cent nickel silver	590	585	588^2	2030	2025	2027^4	2095	2080	2089^4
81-4-15 silicon brass	...	...	...	1510	1510	1510^2	1685	1680	1683^2
65 000 psi manganese bronze[c]	...	...	...	1590	1576	1583^2	1627	1605	1616^2
110 000 psi manganese bronze	...	...	...	1625	1625	1625^2	1695	1690	1693^2

[a] Standard deviation of the average value is ±3 F for all the alloys except the 65 000 psi manganese bronze. For the 65 000 psi manganese bronze alloy, the standard error is ±5 F.

[b] Superscript numerals give the number of specimens.

[c] Data obtained on specimens from lot A.

Patternmaker's Shrinkage:

The previous paper (1) described the details of how the patternmaker's shrinkage was measured by a modification of a method developed by Pilling and Kihlgren (2). The results obtained on castings made in triplicate are presented in Table VI. Gage length of the casting, within the limits of 3 to 9 in., had little effect on the patternmaker's shrinkage of the 76–2½–6½–15 alloy and the nickel silver alloy. For the other three alloys, the patternmaker's shrinkage showed a decided trend to decrease with increase in gage length. The values for the 76–2½–6½–15 alloy and the nickel silver alloy were appreciably lower than those for the other three alloys.

Density and Specific Gravity:

Density measurements were made on machined tension-bar specimens before the bars were tension tested. Details for conducting the measurements with a carbon tetrachloride bath were given previously (1). Accuracy of the values reported in Table VII are estimated to be within < ±0.1 per cent (±0.006 g per cu cm). The values for specific gravity in Table VII were obtained by dividing the density value of the specimens by the density of water, which is 0.998405 g per cu cm at 68 F.

Electrical Resistivity:

The voltage drop was measured with pointed probes across a 3-in. span of

TABLE VI.—PATTERNMAKER'S SHRINKAGE.

Pouring Temperature, deg Fahr	Gage Length, in.	Average Shrinkage, in. per ft[a]	Patternmaker's Shrinkage, in. per ft
76-2½-6½-15 ALLOY			
1810. .	3	0.168	11/64
	6	0.156	5/32
	9	0.180	3/16
		Average 0.168	11/64 ± 1/32
20 PER CENT NICKEL SILVER ALLOY			
2100. .	3	0.108	7/64
	6	0.120	1/64
	9	0.108	7/64
		Average 0.111	1/8 ± 1/32
81-4-15 SILICON BRASS ALLOY			
1690. .	3	0.276	9/32
	6	0.216	7/32
	9	0.204	13/64
		Average 0.232	15/64 ± 1/32
65 000 PSI MANGANESE BRONZE[b]			
1665. .	3	0.252	1/4
	6	0.240	15/64
	9	0.240	15/64
		Average 0.244	15/64 ± 1/32
110 000 PSI MANGANESE BRONZE			
1720. .	3	0.324	21/64
	6	0.262	17/64
	9	0.253	1/4
		Average 0.281	9/32 ± 1/32

[a] Standard deviation of the average values is ±0.007 in. per ft for all of the alloys except the 65 000 psi manganese bronze alloy. For the 65 000 psi manganese bronze alloy, the standard error is ±0.024 in. per ft.

[b] Data obtain on specimens from lot A.

specimens measuring $\frac{3}{8}$ in. in diameter and 6 in. long.[12] Details of the procedure were described earlier (1).

Values for the electrical resistivity of all the alloys except the 65,000 psi manganese bronze at temperatures ranging from 68 to 550 F are given in Table VIII. The temperature range for the 65,000 psi manganese bronze is from 68 to 450 F. The values, estimated to be accurate to within ±0.5 per cent, show that resistivity increases slightly as the temperature is increased. Resistivity is lowest in the 76–2½–6½–15 alloy and 65,000 psi manganese bronze, and is highest in the nickel silver alloy. The electrical conductivities expressed as a per cent of the International Annealed Copper Standard are also presented in Table VIII.

Thermal Conductivity:

The apparatus was essentially the same as the one described by Van Dusen and Shelton (5). Details of the technique were the same as those described in an earlier paper (1).

The values obtained for thermal conductivity are given in Table IX and are estimated to be accurate to within ±5 per cent. The nickel silver, silicon brass and 110,000 psi manganese bronze were about equal in conductivity and lower than the other two alloys which were also about equal to each other. As the temperature increased, the conductivity of each of the alloys increased.

Thermal Expansion:

Cylindrical specimens, $\frac{3}{8}$ in. in diameter and 3 in. long were heated in a vertical position in an electric resistance furnace. A quartz rod resting on the specimen actuated a dial indicator which was sensitive to 0.0001 in. Suitable insulation was used instead of a vacuum in the furnace (1) to minimize the development of convection currents which could result in temperature gradients in the specimen and errors in the measurements.

---

[12] Standard Method of Test for Resistivity of Electrical Conductor Materials (B 193 – 59), 1959 Supplement to Book of ASTM Standards, Part 3, p. 455.

TABLE VII. DENSITY AND SPECIFIC GRAVITY.

Alloy	Density, lb per cu in.			Specific Gravity		
	Max	Min	Avg[a]	Max	Min	Avg
76-2½-6½-15	0.317	0.316	0.317	8.78	8.76	8.77
20 per cent nickel silver	0.321	0.321	0.321	8.90	8.89	8.90
81-4-15 silicon brass	0.299	0.299	0.299	8.27	8.27	8.27
65 000 psi manganese bronze (lot B)	0.301	0.300	0.301	8.33	8.31	8.32
110 000 psi manganese bronze	0.277	0.276	0.277	7.67	7.64	7.66

[a] Average value for three specimens. Standard deviation for the average values is ±0.00022 lb per cu in.

TABLE VIII.—ELECTRICAL RESISTIVITY AND CONDUCTIVITY AT VARIOUS TEMPERATURE LEVELS.[a]

Temperature, deg Fahr	76-2½-6½-15		20 per cent Nickel Silver		81-4-15 Silicon Brass		Manganese Bronze			
							65 000[c] psi		110 000 psi	
	Electrical Resistivity, microhm-cm	Electrical Conductivity, per cent of IACS[b]	Electrical Resistivity, microhm-cm	Electrical Conductivity, per cent of IACS[b]	Electrical Resistivity, microhm-cm	Electrical Conductivity, per cent of IACS[b]	Electrical Resistivity, microhm-cm	Electrical Conductivity, per cent of IACS[b]	Electrical Resistivity, microhm-cm	Electrical Conductivity, per cent of IACS[b]
68	10.4	16.4	36.0	4.8	28.4	6.1	8.4	20.6	21.5	8.0
100	10.6	16.3	36.2	4.8	28.7	6.0	...	...	21.8	7.9
150	10.9	15.8	36.5	4.7	29.0	5.9	8.9	19.3	22.3	7.7
200	11.2	15.4	36.8	4.7	29.4	5.9	9.3	18.5	22.8	7.6
250	11.4	15.1	37.1	4.7	29.7	5.8	9.7	17.8	23.3	7.4
300	11.7	14.7	37.5	4.6	30.1	5.7	10.1	17.1	23.8	7.2
350	12.0	14.4	37.8	4.6	30.5	5.7	10.5	16.5	24.3	7.1
400	12.2	14.1	38.1	4.5	30.9	5.6	10.9	15.8	24.9	6.9
450	12.5	13.8	38.4	4.5	31.4	5.5	11.3	15.3	25.4	6.8
500	12.8	13.5	38.7	4.5	31.9	5.5	...	...	26.0	6.6
550	13.1	13.2	39.0	4.4	32.4	5.3	...	...	26.5	6.5

[a] Only one specimen was tested for each alloy.
[b] The resistivity of the International Annealed Copper Standard is 1.7241 microhm-cm.
[c] Data obtained on specimens from lot A.

TABLE IX.—THERMAL CONDUCTIVITY AT VARIOUS TEMPERATURES.[a]

Temperature, deg Fahr	76-2½-6½-15		20 per cent Nickel Silver		81-4-15 Silicon Brass		Manganese Bronze			
							65,000[b] psi		110,000 psi	
	Btu ft per hr sq ft deg Fahr	Per cent of Copper	Btu ft per hr sq ft deg Fahr	Per cent of Copper	Btu ft per hr sq ft deg Fahr	Per cent of Copper	Btu ft per hr sq ft deg Fahr	Per cent of Copper	Btu ft per hr sq ft deg Fahr	Per cent of Copper
68	41.9	19.0	13.3	6.0	15.3	6.9	50.3	22.1	20.6	9.3
100	43.2	19.6	14.2	6.5	16.0	7.3	52.5	23.1	21.5	9.8
150	45.3	20.7	15.5	7.1	17.2	7.9	55.3	24.3	22.9	10.5
200	47.5	21.8	16.6	7.6	18.3	8.4	57.3	25.2	24.4	11.2
250	49.5	22.9	17.5	8.1	19.4	9.0	58.7	25.8	25.8	11.9
300	51.6	24.9	18.4	8.6	20.6	9.6	60.0	26.4	27.2	12.7
350	53.7	25.2	19.0	8.9	21.7	10.2	61.2	26.9	28.7	13.5
400	55.8	26.3	19.6	9.2	22.8	10.8	62.3	27.4	30.1	14.2
450	57.9	27.4	20.2	9.6	24.0	11.4	64.1	28.2	31.6	15.0
500	60.0	28.6	20.7	9.9	25.1	12.0	...	...	33.0	15.7
550	62.0	29.8	21.2	10.2	26.2	12.6	...	...	34.4	16.5

[a] Only one specimen was tested for each alloy.
[b] Data obtained on specimens from lot A.

Table X gives the mean linear thermal expansion coefficients of each alloy over the temperature ranges of 68 to 100 F and 68 to 550 F. These coefficients are estimated to be accurate to within ±2 per cent. Each alloy was remarkably similar to the others, although the nickel silver had slightly lower values than the others. The coefficient of expansion increased moderately for each alloy with increase in temperature range.

TABLE X.—MEAN LINEAR THERMAL EXPANSION COEFFICIENTS FOR VARIOUS TEMPERATURE RANGES.[a]

Temperature Range, deg Fahr	Coefficient, microinch per in. per deg Fahr				
	76-2½-6½-15	20 per cent Nickel Silver	81-4-15 Silicon Brass	Manganese Bronze	
				65 000 psi[b]	110 000 psi
68 to 100.....	10.2	8.1	9.5	...	10.5
68 to 150.....	10.2	8.4	9.7	10.0	10.7
68 to 200.....	10.3	8.6	9.9	10.2	10.9
68 to 250.....	10.3	8.8	10.2	10.4	11.2
68 to 300.....	10.3	8.9	10.4	10.7	11.4
68 to 350.....	10.4	9.0	10.5	10.9	11.6
68 to 400.....	10.4	9.1	10.6	11.1	11.8
68 to 450.....	10.4	9.2	10.8	11.3	11.9
68 to 500.....	10.4	9.3	10.9	...	11.9
68 to 550.....	10.5	9.4	11.1	...	12.0

[a] Only one specimen was tested for each alloy.

[b] Data obtained on specimen from lot A.

Acknowledgment:

The authors are grateful to the Brass and Bronze Ingot Inst., the sponsor of this program, for permission to publish the data. Appreciation is expressed to the various members of the Brass and Bronze Ingot Inst. for supplying the ingot metal and for conducting the analytical work for chemical composition of the castings. Battelle Memorial Inst. staff members who conducted specialized tasks were W. H. Johnson—melting, casting, measurement of patternmaker's shrinkage, impact properties, hardness measurements, and density measurements; J. E. Campbell—tensile and compression properties; L. G. Beall—fatigue properties; A. Gerds—machinability determinations; A. A. Bauer—melting-range determinations; H. W. Deem—measurements of electrical resistivity, thermal conductivity, and thermal expansion properties; G. Beatty—statistical treatment of the data; and C. H. Lorig—consultant and guidance.

Erratum:

In the previous paper (1), the hardness data were inadvertently interchanged for the 80–10–10 alloy and the 85–5–5–5 alloy in Fig. 5.

REFERENCES

(1) J. G. Kura and R. M. Lang, "Mechanica- and Physical Properties of Three Low-Shrinkage, Copper-Base Casting Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, pp. 775–790 (1958).

(2) J. P. Pilling and T. E. Kihlgren, "A Method for Study of Shrinkage and Its Distribution in Castings," *Transactions*, Am. Foundrymen's Soc., Vol. 40, pp. 210–216 (1932).

(3) A. Brownlee, "Industrial Experimentation," Chemical Publishing Co., Inc., Brooklyn, N. Y., pp. 35–36, Second Edition (1948).

(4) H. W. Gould and K. W. Ray, "Effects of Silicon on the Properties of Brass," *Metals and Alloys*, Vol. 1, April 30, 1930, pp. 455–457.

(5) M. S. Van Dusen and S. M. Shelton, "Apparatus for Measuring Thermal Conductivity of Metals up to 600 C," *Journal of Research*, Nat. Bureau Standards, Vol. 12, pp. 429–440 (1934).

DISCUSSION

MR. N. C. STENGEL.[1]—Why did the longer shake-out time give a change in tensile properties? Is this related to some precipitation that takes place?

MR. W. H. JOHNSON (*author*).—The question refers to the nickel-silver alloy. It is known that wrought nickel-silver alloys are age hardenable. Therefore, it is presumed that the slower cooling associated with the longer shake-out time for the cast nickel-silver alloy resulted in some precipitation hardening which increased the strength and decreased the ductility.

---

[1] Raw Materials Engineer, Western Electric Co., Inc., Allentown, Pa.

STRUCTURAL BERYLLIUM*

By B. B. Muvdi[1]

Synopsis

An attempt was made to produce beryllium possessing adequate structural characteristics. Specimens of plate beryllium representing five different fabrication methods were compared by metallographic examination, texture analysis, and mechanical tests. From a structural viewpoint, the best combination of uniaxial and biaxial properties was exhibited by commercial grade QMV beryllium powder which had been compacted cold and then upset hot. A further advantage of this process over the other four is the fact that it is relatively simple and economical and yields comparatively good surface finish.

The mechanical tests indicated that, for upset beryllium, plastic deformation before fracture was appreciable in both tension and flexure—and even under biaxial stress conditions. The fatigue strength, measured by the cantilever method, was approximately 45 per cent of the tensile strength at 10^7 cycles.

Interest in developing beryllium for structural applications, particularly in the aircraft industry, has been spreading rapidly throughout the country. The reasons for this interest may be found in the outstanding physical, mechanical, and thermal characteristics of beryllium.

Among the physical and mechanical properties that make beryllium attractive to the aircraft designer are its light weight (0.0658 lb per cu in.), its high modulus of elasticity (40 to 45 $\times 10^6$ psi), and relatively high tensile strength (approximately 70,000 to 80,000 psi). The uniqueness of beryllium is partly due to its relatively high melting point (2340 ± 70 F) which renders this metal useful over a wide range of temperatures. Several comparative studies have been made which have demonstrated the superiority of beryllium over the more conventional structural alloys through a wide range of temperatures (**1**, **2**, **3**).[2]

The interest in beryllium has been further enhanced by its attractive thermal properties. Its high thermal conductivity (comparable to that of aluminum) tends to reduce transient thermal gradients. Its low coefficient of thermal expansion (approximately one half that of aluminum) tends to alleviate excessive dimensional growth and related thermal stresses. Its high heat capacity makes beryllium extremely desirable as heat-sink material (**4**). Aside from these unusual characteristics, beryllium possesses extremely good corrosion and oxidation resistance in air at room and elevated temperatures (5).

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Assistant Professor, Department of Engineering Mechanics, Michigan College of Mining and Technology, Houghton, Mich. The work presented here was performed while the author was affiliated with the Martin Co., Baltimore, Md.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The major factors that have contributed to the slow development of beryllium for structural use have been the problems of toxicity, scarcity, and brittleness. While the toxic properties of beryllium constitute a health hazard, the problem is not insurmountable. Protective measures have been devised and beryllium is now produced with a safety record comparable to that of other industries. Insofar as the scarcity of beryllium is concerned, the consensus of the beryllium industry indicates that the occurrence of this metal is adequate for discriminate use in specialized applications. The problem of brittleness, and associated notch sensitivity, has remained unsolved despite the efforts of several investigators (6, 7, 8). Beryllium sheet has been produced which exhibited elongations between 30 to 40 per cent in any one direction in the plane of the sheet (9). However, the fact that beryllium has been made to exhibit appreciable uniaxial ductilities does not render this material structurally useful. To meet the requirements of airframe design, a material should be capable of deforming plastically even under complex stress conditions. Beryllium has been observed in the past to be deficient in this respect. This behavior has been attributed to the preferred crystal orientation resulting from working the metal (6, 8). In the case of cross rolling a plate or sheet, for example, the hexagonal beryllium crystal is discretely oriented to allow slip in the plane of the plate or sheet and very little or no slip in the thickness direction. It was reasoned, therefore, that the multi-axial properties of beryllium could be greatly improved by inducing a random, rather than a preferred, crystallographic texture.

Recognizing the potentialities of beryllium in the airframe industry, the Air Force issued a contract to the Martin Co. to investigate the possibilities of using beryllium in structural application.

Before producing a beryllium structure, however, it was thought advisable to conduct a brief examination of several fabrication processes in order to select the process that would yield optimum characteristics.

In this paper, the several fabrication processes investigated are described. The results of tests performed on specimens prepared by means of the various processes are discussed and evaluated.

Fabrication Techniques

Five different fabrication techniques as illustrated in Fig. 1 were examined in this study. In all instances, the starting billet was prepared by cold compacting the QMV powder[3] into a cylindrical steel container to densities ranging between 70 and 80 per cent of the theoretical value. A brief description of the various fabrication processes follows. A more detailed description of these processes, however, may be found in reference (9).

Hot Pressing:

The cold-compacted beryllium in its container (billet) was soaked at 1850 F for 1 hr in an inert atmosphere. While at this temperature, the beryllium was pressed (compacted) in its container until a density of 100 per cent of theoretical was obtained.

---

[3] The chemical analysis of this powder as supplied by The Brush Beryllium Co. was as follows: beryllium oxide, 0.880 per cent; iron, 0.078 per cent; aluminum, 0.060 per cent; manganese, 0.011 per cent; nickel, 0.013 per cent; magnesium, 0.020 per cent; chromium, 0.013 per cent; and boron, 0.00006 per cent. QMV is The Brush Beryllium Co.'s designation for −200 mesh beryllium powder made by their process from vacuum-melted ingots.

Random Rolling:

The beryllium billet was soaked at 1850 F in an inert atmosphere for 1 hr and passed through rolls in a random fashion. After several passes the finished product was a circular plate with a density of 100 per cent of theoretical and a thickness ranging between 0.2 and 0.3 in.

Hot Pressing Plus Random Rolling:

The beryllium billet at 1850 F was subjected first to hot pressing and then to random rolling. The material reached a density of 100 per cent of theoretical, having a thickness similar to that obtained by random rolling.

Extruding Plus Cross Rolling:

The beryllium billet was soaked at 1850 F for 1 hr, and while at this temperature was extruded using a reduction ratio of 12.5:1 through either square ($1\frac{1}{2}$ by $1\frac{1}{2}$ in.) or rectangular ($\frac{3}{4}$ by $2\frac{3}{4}$ in.) dies. The extrusions were then cut up into 6 in. long pieces and encapsulated in steel frames. The encapsulated beryllium part was then heated to 1850 F and passed through rolls in a direction perpendicular to that of extrusion. Several reduction ratios were used and in all instances the resulting material was 100 per cent of theoretical density and had a thickness comparable to that obtained by random rolling.

Upsetting:

The beryllium billet was soaked at 1850 F for approximately 2 hr after which it was sandwiched between two ground and lubricated platens and upset in a press into a thin circular plate having a density of 100 per cent of theoretical and a thickness ranging between 0.2 and 0.3 in. The upsetting operation was performed as quickly as was practicable in order to minimize heat losses from the billet. Two reduction ratios, 2.4:1 and 6:1, were used in this study.

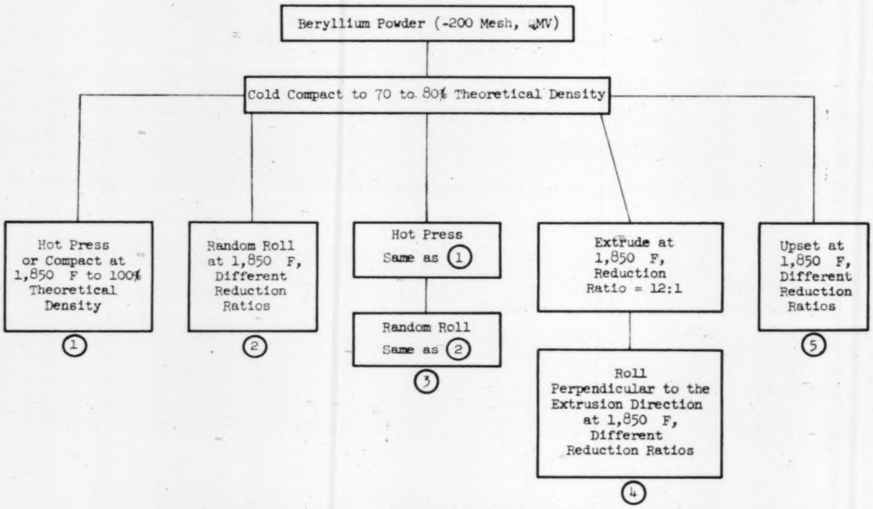

Beryllium Powder (-200 Mesh, QMV)
Cold Compact to 70 to 80% Theoretical Density
Hot Press or Compact at 1,850 F to 100% Theoretical Density
①
Random Roll at 1,850 F, Different Reduction Ratios
②
Hot Press Same as ①
Random Roll Same as ②
③
Extrude at 1,850 F, Reduction Ratio = 12:1
Roll Perpendicular to the Extrusion Direction at 1,850 F, Different Reduction Ratios
④
Upset at 1,850 F, Different Reduction Ratios
⑤

FIG. 1.—Flow Sheet for the Five Fabrication Processes Used.

Test Specimen Preparation:

Several small slices for metallographic examination, $\frac{1}{4}$-in. cubes for X-ray texture analysis, uniaxial tension specimens, and biaxial test specimens were prepared using each of the fabrication techniques described above. The specimen blank was ground flat and, whenever needed, holes were drilled with a carbide drill having a 90 deg tip. The specimens so prepared were then annealed at 1380 F for 2 hr. Approximately 0.005 in. was then removed from each specimen by etching in a 10 per cent aqueous solution of sulfuric acid in order to remove all surface twins remaining as a result of the grinding and other machining operations.

EVALUATION OF PROCESSES

Metallographic Examinations:

Metallographic specimens were cut in the plane of the beryllium plate and transverse to it, then mounted and polished. Photographs were taken of each specimen under polarized light at 150 ×.

A grain size study of specimens prepared by the various fabrication techniques revealed that specimens produced by extruding plus cross rolling possessed the smallest grain size and that specimens produced by random rolling exhibited the largest grain size. However, in all instances, grain sizes were of the same order of magnitude.

Texture Analysis:

A modified Schultz technique was used for the X-ray texture analysis (8, 10). Pole figures were produced by reflecting X-ray beams off three sides of a $\frac{1}{4}$-in. cube. Copper K_α radiation at a maximum of 40 kv and 20 ma was used in combination with a 1 or 4 deg divergence slit. All specimens were rotated throughout 360 deg, and X-ray scans in steps of at least 10 deg were taken from the center of the pole figure to the outer edge.

Examination of the pole figures for the material produced by the various processes indicated that hot pressing yielded the most nearly random texture, while extruding plus cross rolling resulted in the highest degree of preferred orientation in the plane of the plate. Material produced by random rolling, hot pressing plus random rolling, and upsetting exhibited some preferred orientation in the plane of the plate, but to a degree much less pronounced than that induced by extruding plus cross rolling.

Uniaxial Tension Testing:

Tension specimens approximately 3 in. long were used in this phase of the investigation. This specimen had a gage length of 1 in., a gage width of 0.300 in. and a gage thickness of 0.270 in. Strain measurements were made by means of SR-4 (type A-7) strain gages mounted on each specimen. All specimens were loaded by means of a jig especially designed to minimize eccentric loading.

Approximately six specimens from each fabrication process were tested. The average of the results indicated that the modulus of elasticity was, for all practical purposes, unaffected by method of fabrication. With regards to the strength characteristics, however, upsetting as well as hot pressing plus random rolling were superior to the other fabrication methods. Material produced by hot pressing yielded the lowest strength values. The highest uniaxial ductility was obtained by extruding plus cross rolling, and the lowest by hot pressing.

Biaxial Testing:

In an effort to determine the strength and ductility properties of beryllium under biaxial stress conditions, and in view of the fact that the only material available was in the form of plate, it was decided to induce biaxial stress condition by means of a concentrated load on a circular plate rigidly fixed around its circumference.

The specimen used as well as the apparatus employed for this test are shown in Figs. 2 and 3. Copper was purposely chosen for the bushings and the tip on the end of the 1-in. diameter ram in order to minimize "Brinelling" action. The diameter of the exposed beryllium plate was 3 in. and its thickness was 0.170 in.

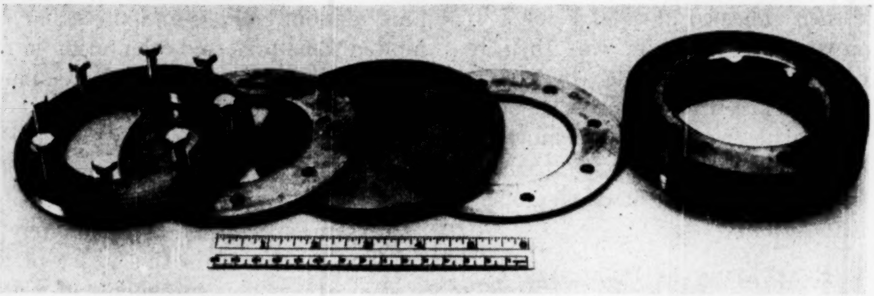


FIG. 2.—Biaxial Tension Test Specimen Holder and Specimen.

Left to right: Top steel ring with aluminum bolts, top copper bushing, beryllium specimen, bottom copper bushing, and bottom steel support ring.

In an effort to measure the strain that developed under biaxial stresses, a strain gage (type SR4-PA-3) was mounted to the bottom center of the plate. The deflection at the center of the plate was measured by means of a dial indicator.

Two specimens prepared by each fabrication process were tested. The results indicated that on the basis of strength and ductility under biaxial stress conditions, the material fabricated by upsetting yielded the best properties, while the poorest properties were obtained from the extruded plus cross-rolled material. The explanation for this difference in behavior may be found in the textures resulting from the two fabrication processes. While upsetting yielded a "semi-random" type of texture (thus enabling slip to occur not only in the plane of the plate but also through the thickness), extruding plus cross rolling resulted in a severe preferred orientation in the plane of the plate which minimized slip through the thickness.

Examination of the deformation pattern of the beryllium sheets subjected to biaxial stressing indicated that the plastic deformation was concentrated in a circle approximately $\frac{3}{8}$ in. in diameter about the center of the plate. The permanent strain recorded (approximately 0.4 to 0.5 per cent for the upset material),

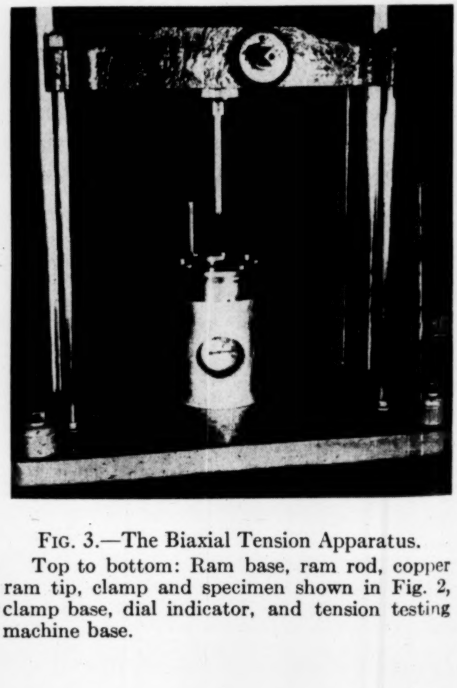


FIG. 3.—The Biaxial Tension Apparatus.

Top to bottom: Ram base, ram rod, copper ram tip, clamp and specimen shown in Fig. 2, clamp base, dial indicator, and tension testing machine base.

however, was measured with a strain gage averaging over a distance of $\frac{3}{4}$ in. This means that the value of strain (0.4 to 0.5 per cent) is actually a very conservative value and that a larger value would have been recorded had the strain gage been averaging over a shorter distance, a distance corresponding to that in which actual plastic deformation occurred.

TABLE I.—MERIT RATING OF SEVERAL BERYLLIUM FABRICATION PROCESSES.

Fabrication Process	Grain Size	Randomness of Texture	Uniaxial Tension Tests		Biaxial Tests		Total
			Strength	Ductility	Strength	Ductility	
Hot pressing	2	1	4	4	3	2	16
Random rolling	4	2	3	2	2	2	15
Hot pressing plus random rolling	3	2	1	2	2	2	12
Extruding plus cross rolling	1	3	2	1	4	3	14
Upsetting	2	2	1	3	1	1	10

Selection of Optimum Process:

A summary of the results of tests performed is given in Table I. This table indicates, for each of the parameters examined, the rating given to each fabrication process in comparison to the others. Number 1 stands for the best, number 2 for the second best, and so on, so that the sum of these numbers for all the parameters investigated in a given process indicates the relative overall merits of this process in comparison with others.

Consideration of this comparative study indicates that relatively better properties can be obtained by upsetting or by hot pressing plus random rolling than by any of the other three processes. While the differences in properties resulting from upsetting and from hot pressing plus random rolling were, for all practical purposes, negligible, the former technique was chosen because it is easier and more economical to perform; it yields a better surface finish and is less likely to produce surface cracking than the latter technique.

In order to evaluate further the mechanical properties obtained by the upsetting process, additional specimens were fabricated using this technique with a reduction ratio of 6:1. These specimens consisted of uniaxial tension, cantilever fatigue, and flexure coupons, the results of which are discussed in the following paragraphs.

ADDITIONAL TESTS ON UPSET BERYLLIUM

Uniaxial Tension Tests:

The tension specimen used in this phase of the program was of the pin-loaded type similar to that described previously except that the gage length was 2 in. and the thickness 0.2 in.

An attempt was made to determine the sensitivity of beryllium tensile properties at room temperature to variations in the strain rate. Since available extensometers were incapable of recording at relatively high rates of straining, SR-4 (type A-5) strain gages were used for the purpose. One strain gage was mounted on each of the wide surfaces of the specimen, and their outputs were separately recorded on a Consolidated recording oscillograph. A 10,000-lb Baldwin SR-4 load cell was used to permit recording of the applied load.

The specimens were tested on a Baldwin FGT universal testing machine having a rated capacity of ± 150,000 lb. In an effort to minimize eccentricity of loading, long linkages with universal joints were used.

The rate of straining was approximately controlled by the machine head travel, and actual strain rate values were subsequently computed from the oscillograph records. Four specimens were tested at each of five strain rates, which varied from 0.001 to 0.050 in. per in. per min. The results of these tests are shown in Table II and illustrate that effect of strain rate on the properties is relatively insignificant. Any variation encountered in the measured values could be attributed to normal scatter of the data. The results also indicate that upset beryllium (reduction ratio = 6:1) possesses the following average tensile properties:

Modulus of elasticity, psi........	41.0 × 10⁶
Proportional limit, psi..........	19 000
0.2 per cent yield strength, psi....	37 000
Ultimate tensile strength, psi.....	71 000
Elongation, per cent............	6.7
Reduction of area, per cent.......	6.4

TABLE II.—RESULTS OF TENSION TESTS AT ROOM TEMPERATURE FOR UPSET BERYLLIUM.

Strain Rate, in. per in. per min	Modulus of Elasticity, psi	Proportional Limit, psi	0.2 per cent Yield Strength, psi	Tensile Strength, psi	Elongation, per cent	Reduction of Area, per cent
0.001......	[a]	[a]	[a]	63.0 × 10^3	3.5	4.4
	[a]	[a]	[a]	73.0	6.0	5.2
	41.0 × 10^6	22.0 × 10^3	38.0 × 10^3	74.0	8.5	8.7
	40.5	18.0	35.0	66.0	4.0	5.5
0.010......	42.0	20.0	39.0	75.0	8.0	5.7
	38.0	22.0	39.0	74.0	8.0	8.0
	39.5	21.0	40.0	70.0	6.0	5.9
	43.0	18.0	36.0	71.0	5.0	6.8
0.020......	39.5	21.0	40.0	76.0	9.0	7.5
	38.0	20.0	36.0	72.0	9.0	8.8
	39.5	22.0	41.0	76.0	9.0	7.9
	42.0	18.0	35.0	70.0	7.0	6.4
0.040......	40.0	17.0	36.0	73.0	7.0	6.4
	43.0	17.0	36.0	71.0	[b]	5.2
	42.5	18.0	35.0	69.0	6.5	5.6
	41.0	18.0	35.0	67.0	4.5	4.5
0.050......	43.0	19.0	36.0	71.0	6.0	6.4
	41.5	18.0	36.0	73.0	7.5	7.4
	40.0	18.0	37.0	68.0	6.0	6.5
	39.5	20.0	37.0	70.0	6.5	6.0
Average.....	41.0	19.0	37.0	71.0	6.7	6.4

[a] No record was obtained because of oscillograph difficulties.

[b] Broke outside gage length.

It is observed that beryllium exhibits a high ratio of ultimate strength to yield strength (approximately 1.9), which is indicative of appreciable strain hardening before fracture. Furthermore, the average elongation observed in this test (6.7 per cent) is comparable to that observed for other structural materials such as high-strength aluminum and precipitation hardening stainless steels. A few typical stress-strain curves for cold compacted and upset beryllium are shown in Fig. 4.

The relatively low value of 0.2 per cent yield strength is a definite disadvantage in any structural application involving upset beryllium. However, there are definite indications that the yield strength may be increased, without appreciably affecting the other properties, by variations in the upsetting or annealing temperatures. Mechanical testing of extruded beryllium has shown that the yield strength increases as the extrusion temperature is lowered. While examination of this effect in the case of the upsetting process is highly desirable, it was beyond the scope of this investigation and future work on beryllium fabrication processes should take it into consideration.

It is worthwhile noting that, while most materials under tensile loading undergo an appreciable amount of "necking" before fracture, the beryllium specimens fractured without any indication of localized "necking." Whatever plastic deformation occurred in the process of straining was distributed uniformly over the entire gage length.

Another interesting observation is the fact that, in all instances, the specimen pinholes deformed plastically to an appreciable extent. This is significant in pointing out that beryllium is capable of localized plastic flow which tends to alleviate stress concentrations at points of discontinuities. Furthermore, this behavior indicates that beryllium may be successfully joined by riveting or bolting.

Flexure Tests:

The flexure specimens were approximately 0.2 by 0.6 by 5.0 in. flats with a free span of 3.0 in. and were loaded in the manner illustrated in Fig. 5. In order to minimize "Brinelling" action, $\frac{1}{4}$-in. diameter supports and a $\frac{1}{4}$-in. diameter ram tip were used. A Baldwin PTE testing machine having a rated capacity of ±5000 lb was employed. The load was gradually applied at the approximate rate of 200 lb per min. The use of a Baldwin PD-1M deflectometer permitted autographic recording of load-deflection curves.

The results of these tests are shown in Table III. The modulus of rupture was calculated using the equation $\sigma =$

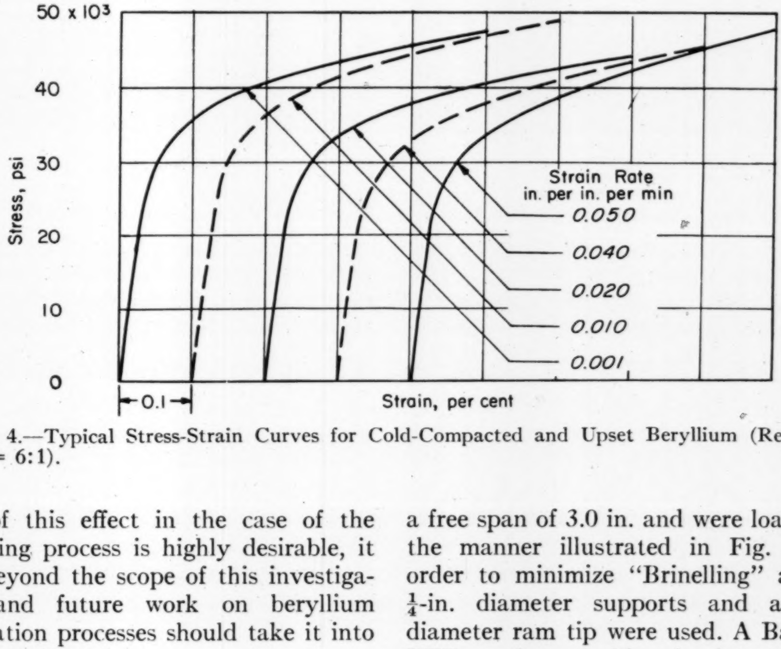


FIG. 4.—Typical Stress-Strain Curves for Cold-Compacted and Upset Beryllium (Reduction Ratio = 6:1).

Mc/I, where σ is the modulus of rupture, M the fracture moment, c the distance from neutral axis to outer fiber, and I the moment of inertia; and the modulus of elasticity was calculated by using the elastic portion of the load-deflection curve and the equation $E = PL^3/48\delta I$, where E is the modulus of elasticity, P the load to produce an elastic deflection δ, and L the free span. An appreciable amount of scatter is observed in these data, particularly in the deflection values.

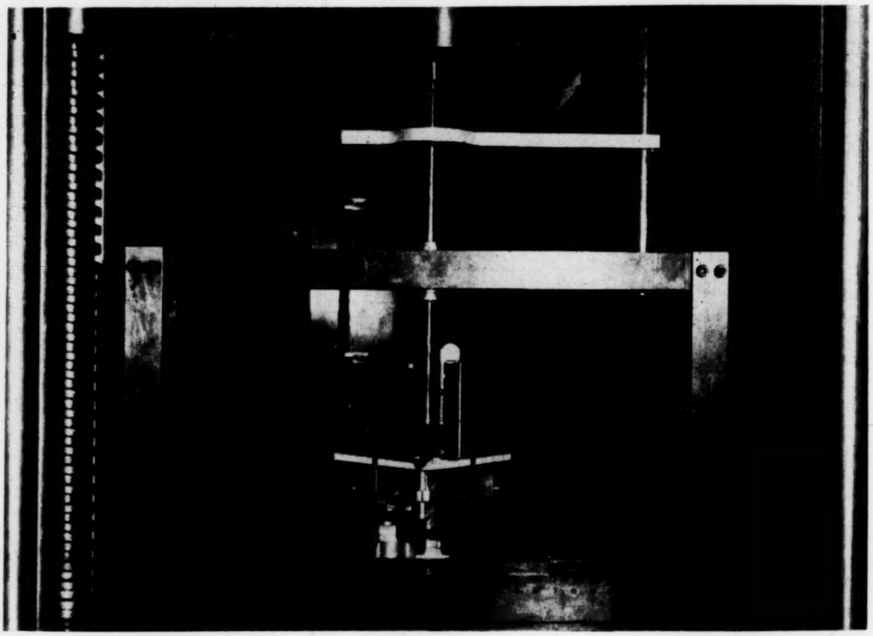


FIG. 5.—Flexure Test Setup

The average modulus of rupture for the six specimens is 114,000 psi which, when divided by the average ultimate tensile strength (71,000 psi), yields a ratio approximately equal to 1.6. This value is approximately the same as the value commonly used in design for ductile materials, namely 1.5.

The average modulus of elasticity value determined in this test (33.3×10^6 psi) is appreciably lower than that obtained in the tension test, namely, 41.0×10^6 psi. The reason for this discrepancy is not evident from the few tests performed.

Cantilever Fatigue Tests:

The cantilever fatigue specimen used in this study is illustrated in Fig. 6. The specimen was designed so that a constant stress was induced along the specimen gage length. The tests were performed on a Sonntag (model SF-1-U) fatigue testing machine, as shown in Fig. 7, using a stress ratio $R = -1.0$. The ends of the specimen were sandwiched in hard rubber cushions in order to minimize the effect of stress concentration due to gripping.

The S-N curve for the specimens tested in this program is shown in Fig. 8. While the number of specimens tested was by no means sufficient to determine accurately the fatigue properties of beryllium, the data obtained did serve to

TABLE III.—RESULTS OF FLEXURE TESTS AT ROOM TEMPERATURE FOR UPSET BERYLLIUM.

Loading rate = 200 lb per min

Specimen Number	Modulus of Rupture, psi	Modulus of Elasticity, psi	Deflection, in.
1..........	108.0 × 10^3	32.5 × 10^6	0.12
2..........	121.0	37.6	0.44
3..........	118.0	31.3	0.34
4..........	100.0	34.0	0.11
5..........	112.0	31.2	0.31
6..........	122.0	33.4	0.51
Average...	114.0	33.3	0.31

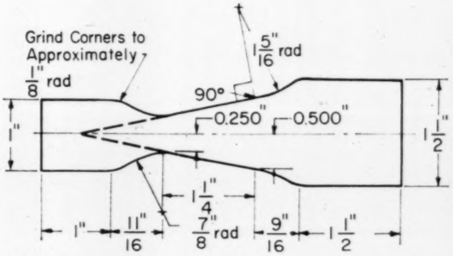


FIG. 6.—Design of Beryllium Cantilever Fatigue Specimen.

NOTE.—All dimensions in inches.

establish the trend needed for evaluating the fatigue behavior of upset beryllium. Examination of the data indicates that beryllium possesses a fatigue strength (at 10^7 cycles) approximately 45 per cent of the ultimate tensile strength. This behavior compares favorably with that of ductile materials such as steels, which exhibit endurance limits approximately 50 per cent of their ultimate tensile strength values. Whether the fatigue strength obtained at 10^7 cycles constitutes a true endurance limit cannot be determined from these data. Further testing is, therefore, needed to establish definitely whether or not the curve continues to descend with further increase in the number of cycles beyond 10^7. However, the trend of the fatigue curve obtained in this program indicates a leveling-off effect beyond 10^6 cycles,

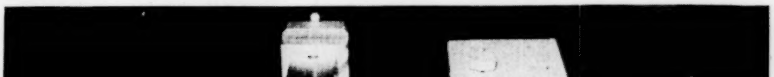


FIG. 7.—Cantilever Fatigue Test Setup.

indicating that beryllium may exhibit a true endurance limit.

CONCLUSION

The results presented in this paper lead to the following conclusions:

1. Of the five fabrication processes examined, upsetting was chosen as the most economical process yielding optimum properties and good surface finish.

2. Upset beryllium (reduction ratio = 6:1) yielded the following tensile properties:

Modulus of elasticity, psi	41.0 × 10^6
Proportional limit, psi	19 000
0.2 per cent yield strength, psi	37 000
Ultimate tensile strength, psi	71 000
Elongation, per cent	6.7
Reduction of area, per cent	6.4

3. The beryllium tension test specimens exhibited uniform plastic deformation along the specimen gage length, resulting in approximately equal values of elongation and reduction of area. Appreciable permanent set was observed at the pin holes, indicating that beryllium is capable of alleviating stress concentrations due to discontinuities. This behavior also indicates that it may be possible to join beryllium to itself or to other materials by bolting or riveting.

4. Upset beryllium (reduction ratio = 6:1) exhibited a fatigue strength at 10^7 cycles equal to approximately 45 per cent of its ultimate tensile strength.

5. Upset beryllium exhibited appreciable plastic deformation even under biaxial stress conditions.

6. Properly fabricated beryllium may be used to produce useful structural components.

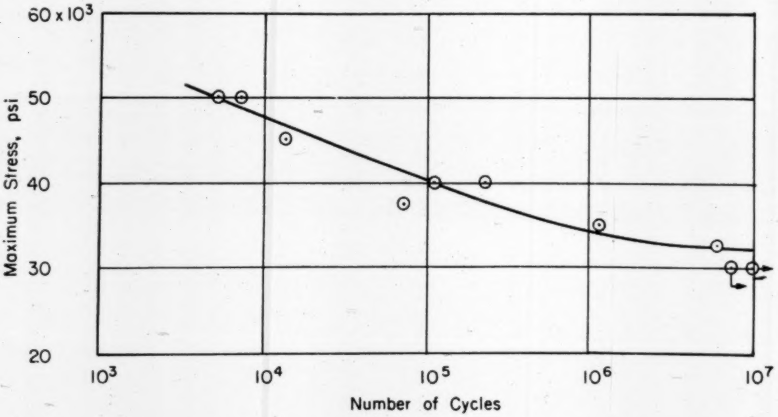


FIG. 8.—*S-N* Curves for Upset Beryllium (Cantilever Type Fatigue, $R = -1.0$).

Acknowledgments:

The work presented in this paper was sponsored by the Aircraft Laboratory, Wright Air Development Center, with Mr. R. D. Guyton as Project Engineer, whose valuable technical assistance during the course of the investigation is greatly appreciated. Credit is due F. M. Yans and A. R. Kaufmann of Nuclear Metals, Inc., who, as subcontractors to the Martin Co., performed and examined the various fabrication processes. Credit is also given to C. J. Giemza of The Martin Co. for his efforts in the initial formulation of the program.

REFERENCES

(1) O. J. Bush, "Beryllium, Its Uses as a Structural Material, Past and Present," The Martin Co., Engineering Report No. 8961 (1956) (SECRET).

(2) R. E. Foster and L. A. Riedinger, "Future Weight Reductions with Beryllium," Lockheed Aircraft Corp., Missile Systems Division, LMSD-2526, Paper 159 (1957).

(3) G. A. Hoffman, "Beryllium as an Aircraft Structural Material," The Rand Corp. (1957).

(4) W. H. Kinard, "The Behavior of Beryllium and Beryllium Copper in a 4000 F Supersonic Air Jet at a Mach Number of 2," National Advisory Committee for Aeronautics, Research Memorandum L57G31 (1957) (CONFIDENTIAL).

(5) W. W. Beaver and K. G. Wikle, "Mechanical Properties of Beryllium Fabricated by Powder Metallurgy," *Journals of Metals*, Am. Inst. Mining and Metallurgical Engineering, Reprint No. 7 (1954).

(6) J. L. Klein, V. Macres, D. H. Woodard, and J. Greenspan, "Ductility of Beryllium as Related to Preferred Orientation and Grain Structure," The Metal Beryllium, Chapter VII-C, Am. Soc. for Metals, Cleveland, Ohio (1955).

(7) G. L. Tuer and A. R. Kaufmann, "Ductility of Beryllium as Related to Single Crystal Deformation and Fracture", in The Metal Beryllium, Chapter VII-B, Am. Soc. for Metals, Cleveland, Ohio (1955).

(8) J. Greenspan, "Texture, Microstructure, Ductility and Other Tensile Properties of Hot Pressed and Hot Rolled Beryllium Powder Compacts," Master Thesis, Nuclear Metals, Inc. (1955).

(9) B. B. Muvdi, "Structural Evaluation of Beryllium Produced by Several Processes," Wright Air Development Center, Technical Report 58-162 (1958).

(10) C. S. Barrett, "The Structure of Metals," McGraw-Hill Book Co., Inc., New York, N. Y., p. 179 (1952).

DISCUSSION

MR. J. C. HAMAKER, JR.[1]—The author has conducted a comprehensive property study of a strategically promising material.

Is there any explanation for the very low proportional limit of beryllium? In the ultra-high-strength steel field, we sometimes obtain proportional limits of 40 per cent of the ultimate tensile strength. In the beryllium data presented here, I believe it was only 25 to 30 per cent of the ultimate strength. Is this low value significant to aircraft design? Is there any reason for it? In the heat-treated steels, it is felt to be associated in some way with the heat treatment, such as residual stresses, undertempering, etc.

Secondly, with regard to the fatigue properties, I believe the author reported an appreciably higher endurance limit, or tendency toward an endurance limit, than the proportional limit. In other words, the actual stressing was considerably above the proportional limit. Comments on this would also be appreciated.

MR. B. B. MUVDI (*author*).—As pointed out by Mr. Hamaker, the ratio of porportional limit to ultimate strength is rather low when compared to the more conventional materials such as steels. Obviously, a low proportional limit is not desirable from a design standpoint. Of more significance in aircraft design, however, is the 0.2 per cent yield strength which, for beryllium, was slightly over 50 per cent of the ultimate strength. Furthermore, it is believed that the yield strength and, possibly, the proportional limit might be improved by proper changes in the processing or annealing temperature, as well as the reduction ratio in the upsetting process.

---

[1] Director of Research and Metallurgical Engineering, Vanadium-Alloys Steel Co., Latrobe, Pa.

The ratio of endurance limit to proportional limit was found to be approximately 1.7. This strengthening effect due to cyclic loading has also been observed with other materials such as steels, particularly when heat treated to low hardness levels. However, the strengthening effect encountered in the case of beryllium is unusually high and no explanation can be offered for this behavior on the basis of the few tests that were performed.

A MEMBER.—Could you tell us what the oxide content of the beryllium material was in your samples? And would you expect an oxide-free material to show improved mechanical physical properties?

MR. MUVDI.—The oxide content of the beryllium used in this investigation was 0.88 per cent. It is believed that beryllium oxide is a factor that contributes to embrittlement. For this reason, efforts were made to secure as low an oxide content as is possible with commercially pure beryllium.

A STUDY OF MECHANICAL BEHAVIOR OF INCONEL 718 ALLOY FOR DESIGN OF A GLIDE VEHICLE

By N. A. Tiner[1] and J. E. Fischler[2]

Synopsis

A study is made of the mechanical behavior of a precipitation strengthened nickel-chromium alloy, Inconel 718, under the temperature-stress-time conditions encountered in flight by a typical hypersonic glide vehicle. The effect of temperature on the coefficient of linear expansion, Young's modulus, tangent modulus, yield strength, and creep resistance is discussed, and the roles of microstructure and precipitation process in certain mechanical behaviors are evaluated. The relation of the mechanical properties to the failure by deformation of the structural components of the glide vehicle is clarified for design purposes.

As a boost glide vehicle travels at hypersonic speeds through the atmosphere, the heat generated at the boundary layer is transmitted from the surface to the interior, and the temperature of the structural components is raised above 1000 F. At these operating temperatures, the materials used for the structure begin to exhibit time-dependent deformation and microstructural changes.

A realistic design of the vehicle should take full advantage of the high-temperature strength characteristics of the material used and overcome the failure of the structural components by excessive deformation due to temperature- and time-dependent changes.

The purpose of this paper is (1) to evaluate the precipitation strengthening and deformation behavior of a nickel-chromium alloy, Inconel 718, developed by the International Nickel Co., and (2) to describe briefly the temperature-stress-time conditions of a typical glide vehicle and the relation of the failure by deformation of its structural components to the mechanical behavior of the alloy. A detailed analysis of the structural design parameters is presented in a technical report to the Wright Air Development Division of Air Research and Development Command (**1**).[3]

TEMPERATURE-STRESS-TIME CONDITIONS IN A HYPERSONIC FLIGHT

A typical hypersonic glide vehicle has a range of about 5000 miles. It is boosted to an altitude of about 200,000 ft in 3 to 4 min. It reaches a speed of approximately Mach 17, glides for about 40 min, and then begins its landing approach from an altitude of about 30,000 ft (Fig. 1).

---

[1] Specialist for Metallurgical Research Projects, Douglas Aircraft Co., Santa Monica, Calif.

[2] Group Leader for Advanced Vehicle Analysis, Douglas Aircraft Co., Santa Monica, Calif.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

During this basic mission trajectory, the structural components of the vehicle are exposed to a period of rapid heating, a period of holding at peak temperature, and a period of cooling. The typical temperature histories for two structural elements on the lower surface of a multispar wing are shown in Fig. 1. One of the elements was taken in the vicinity of the leading edge near the intersection with fuselage and the other at a point $\frac{1}{2}$ chord distance from the leading edge. The temperatures were calculated by numerical analysis on an IBM computer. The transient heat transfer by conduction, convection, and radiation was included in the program. These temperatures represent nearly maximum and intermediate temperatures for the structural components of the entire wing (1).

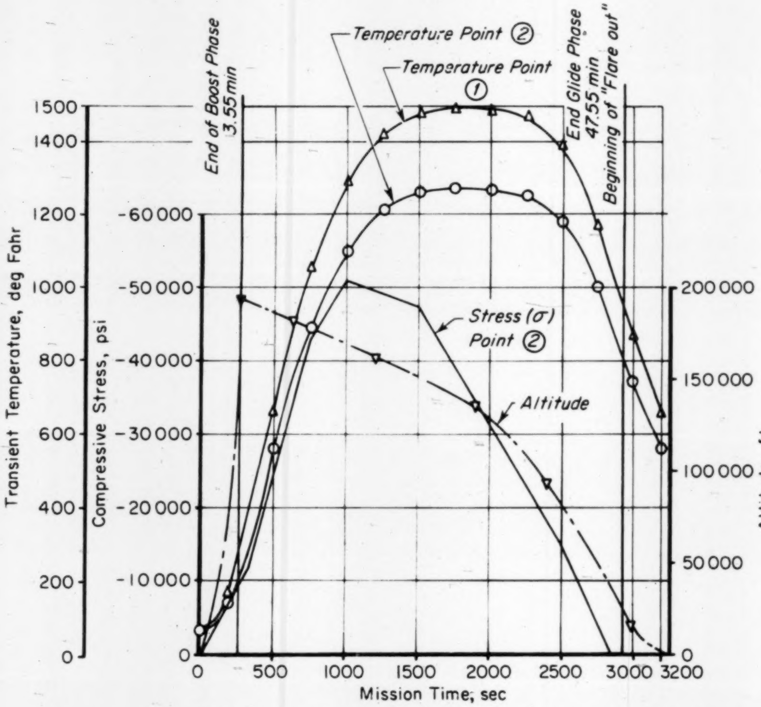


FIG. 1.—Basic Flight Mission for a Typical Hypersonic Boost Glide Vehicle.

Stress-temperature-time history for a spar cap on the lower wing surface at two points near the intersection with the fuselage, point (1) near the leading edge, point (2) $\frac{1}{2}$ chord length from the leading edge.

The average principal stresses developed during flight on the lower spar cap at the point $\frac{1}{2}$ chord distance from the leading edge are also indicated in Fig. 1. The stresses developed in the vicinity of the leading edge were comparatively small and are due mainly to the high thermal gradients in the structure. The load stresses resulting from supporting the weight of the vehicle are also very small.

A number of perturbations may occur from the basic mission trajectory as, for example, gusts, wind shear, and planned maneuver. The effect of a typical planned maneuver (360-deg turn) on the temperature and stress exposure histories is illustrated in Fig. 2, which represents a very severe perturbation.

These typical temperature, stress, and time conditions of flight constitute the broad environmental requirements for the design of the multispar wing components of a glide vehicle. The mechanical behavior of Inconel 718 alloy will be evaluated in the light of these requirements.

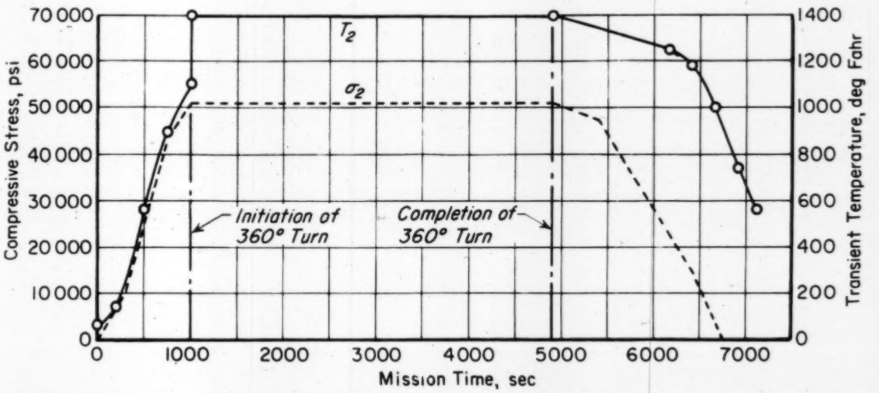


FIG. 2.—Stress-Temperature-Time History for 360-deg Turn Perturbation to Basic Mission Same structural element as point (2) in Fig. 1.

EXPERIMENTAL OBSERVATIONS ON THE BEHAVIOR OF INCONEL 718 ALLOY

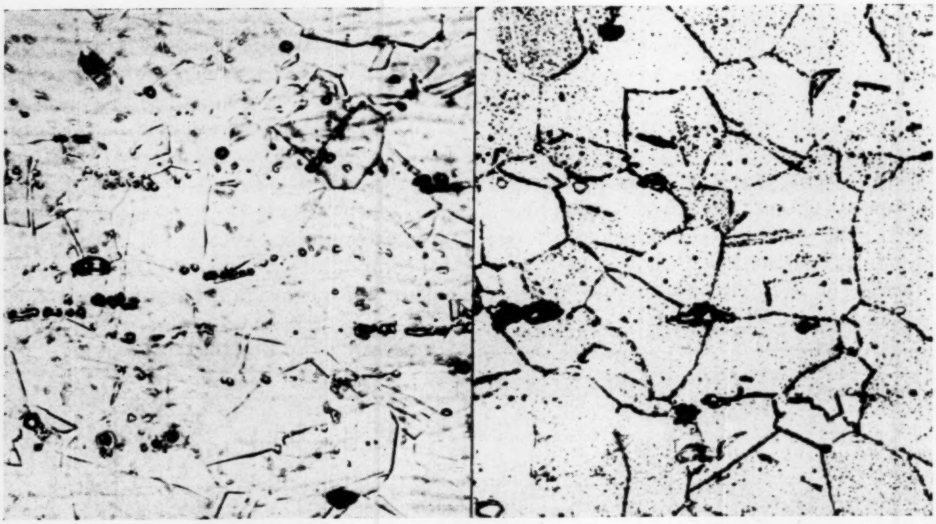
General Microstructural and Strengthening Characteristics:

Inconel 718 alloy developed by the International Nickel Co. has a typical composition of 18.0 per cent chromium, 17.7 per cent iron, 4.6 per cent columbium and tantalum, 2.9 per cent molybdenum, 0.8 per cent titanium, 0.4 per cent aluminum, and 0.05 per cent carbon. It consists essentially of a Ni-Cr-Fe solid solution matrix and dispersion of second phase particles. The balance between the matrix and the dispersed particles is achieved by subjecting the alloy to a sequence of heating and cooling cycles. Solution annealing at a temperature of 1800 F produces a matrix of polygonal grains of gamma phase and a number of dispersed particles of CbN. The gamma phase has a face-centered cubic structure of Ni-Cr-Fe and contains atoms of other elements such as molybdenum, columbium, tantalum, titanium, and aluminum. Aging at the temperatures of 1000 to 1500 F strengthens the alloy through precipitation of intermetallic compounds of the $Ni_3(Al,Ti)$ type within the grains and complex carbides and nitrides at the grain boundaries, as noted by other investigators (**3–7**) in similar nickel alloys (Figs. 3 and 4).

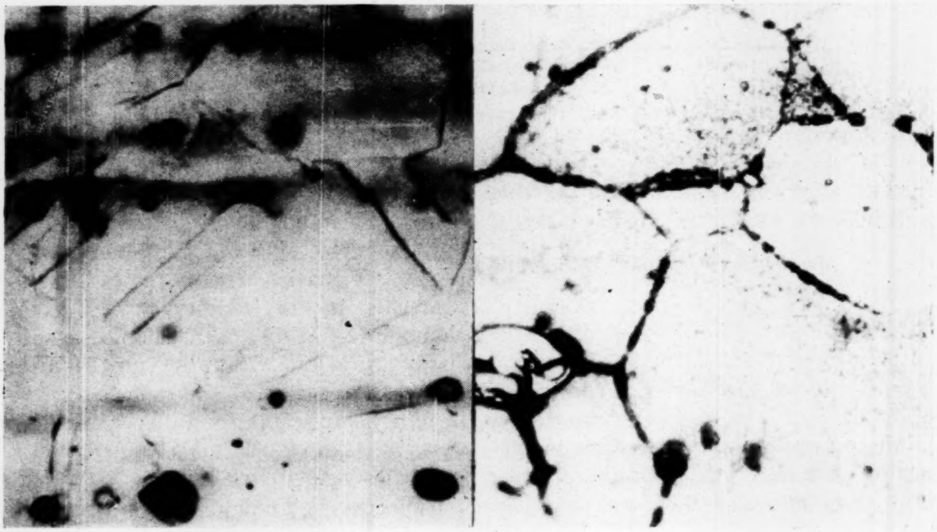
The formation of the second phase particles in the vicinity of the grain boundaries, in the matrix along the subboundaries, and within the substructures appears to occur in one stage, and the strengthening appears to be associated with the coherency of the precipitate particles (**8,9**).

The strengthening by precipitation may be examined in detail by heating a series of specimens of Inconel 718 alloy at different temperatures for various periods of time, water quenching, and determining the changes in hardness at room temperature. The results of such determinations are shown in Fig. 5.

The curves appeared to follow the ex-

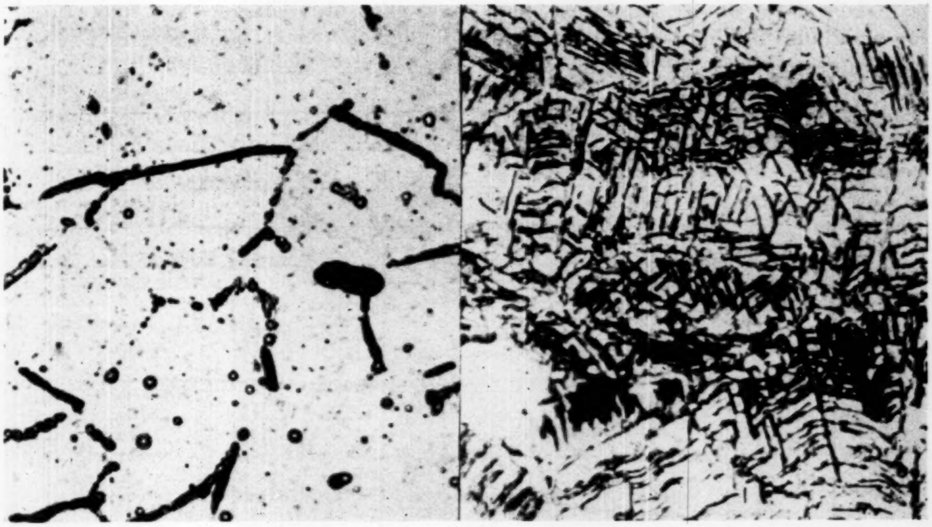


(*a*) Mill annealed (×500). (*b*) Mill annealed (×1000).



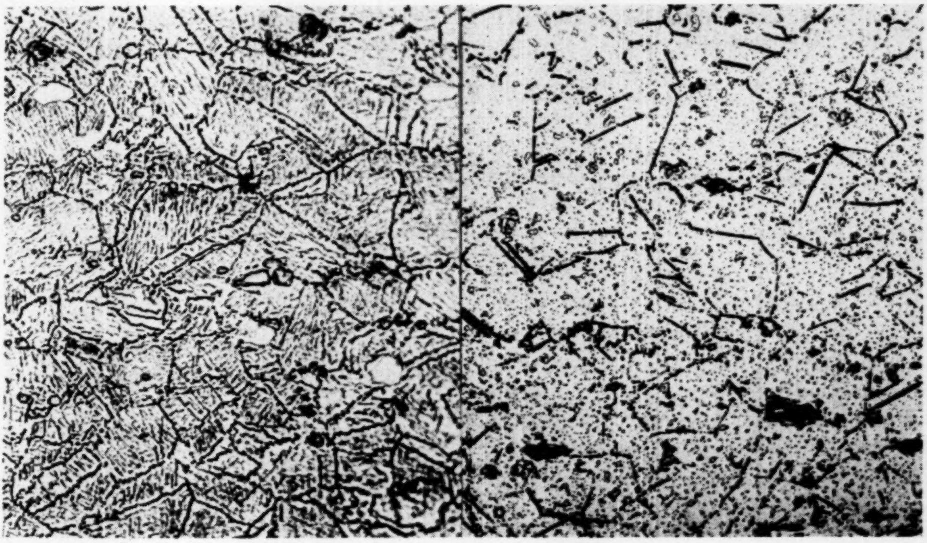
(*c*) Mill annealed and aged at 1300 to 1320 F for 16 hr (×500). (*d*) Mill annealed and aged at 1300 to 1320 F for 16 hr (×1000).

FIG. 3.—Photomicrographs Showing Effect of Mill Annealing and Aging on Microstructure of Inconel 718 Alloy. Specimens electropolished and etched with modified aqua regia.



(*a*) Mill annealed and aged at 1500 F for ½ hr (×500).

(*b*) Mill annealed and aged at 1500 F for ½ hr (×1000).



c) Mill annealed and aged at 1500 to 1530 F for 100 hr (×500).

(*d*) Mill annealed and aged at 1500 to 1530 F for 100 hr (×1000).

FIG. 4.—Photomicrographs Showing Effect of Overaging on Microstructure of Inconel 718 Alloy. Specimens electropolished and etched with modified aqua regia.

pected behavior of age hardening, with the maximum hardness and the time to maximum hardness decreasing as the aging temperature was increased. No multiple hardness peaks were noted. The greatest hardening was achieved at 1200 F; exposures above this temperature tended to reduce hardening and caused overaging. The time needed to reach the maximum hardness attainable at a given temperature was shortest at about 1500 F.

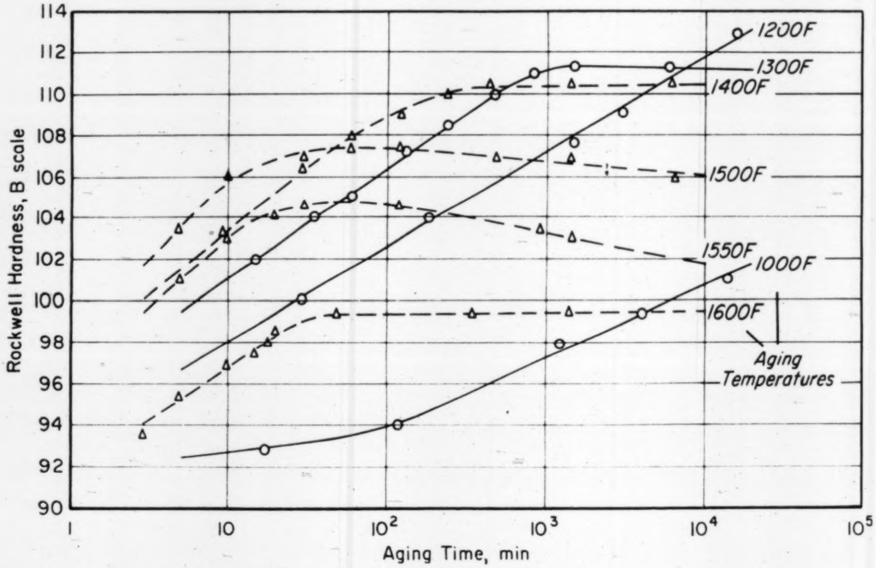


FIG. 5.—Effect of Aging Temperature and Time on Room Temperature Hardness of Incone 718 Alloy (Mill Annealed).

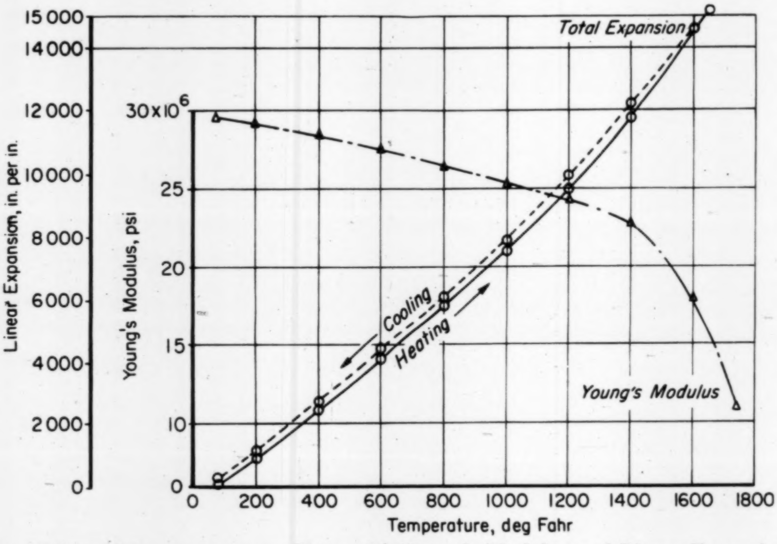


FIG. 6.—Effect of Temperature on (Dynamic) Young's Modulus and Linear Expansion of Inconel 718 Alloy. Aged 16 hr at 1300 F.

From the foregoing analysis of the precipitation hardening behavior, it can be concluded that an aging treatment of 16 hr in the vicinity of 1300 F would produce a hardness close to maximum in a reasonably short period of time. This aging treatment was considered a suitable initial condition for the structural components in hypersonic flight.

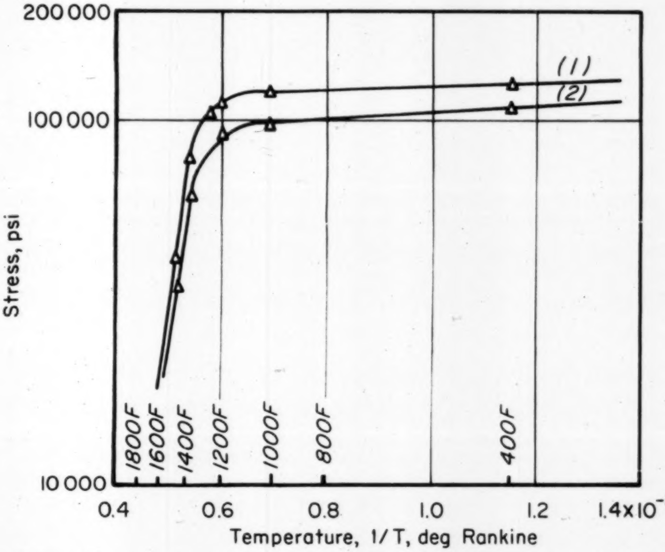


FIG. 7.—Effect of Temperature on 0.2 per cent Offset Yield Strength and Proportional Limit of Inconel 718 Alloy Sheet.

Aged at 1300 to 1320 F for 16 hr and tested at 0.1 in. per in. per min strain rate. (1) 0.2 per cent offset yield strength; (2) proportional limit.

Deformation Characteristics:

The typical variations of the thermal expansion coefficient and Young's modulus with temperature are shown in Fig. 6. The measurement of thermal expansion was made with a fused quartz tube dilatometer, and that of Young's modulus with the dynamic method (**2**). The curves showed a rather noticeable change in curvature at the the temperatures of 1300 to 1400 F, presumably caused by precipitation reactions.

The variations of the log of stress at the proportional limit and at the 0.2 per cent offset yield with the reciprocal of temperature are shown in Fig. 7. The stress values were determined from the short-time tension load-strain curves, which were obtained by means of a Riehle universal testing machine by heating the specimens to the desired temperature in 10 min, soaking for 5 min, and subsequently straining at a constant rate of 0.1 in. per in. per min.

The log-stress versus reciprocal temperature curve consisted of two rectilinear branches of different slope and a transition line connecting them in the temperature range of 1200 to 1400 F. This behavior is generally associated with two different deformation mechanisms with different activation energies (**10,11**). It appears that the rate processes controlling the deformation of Inconel 718 at low temperatures begin to break down at about 1200 F, and the high-temperature deformation mechanism becomes operative at temperatures above 1400 F.

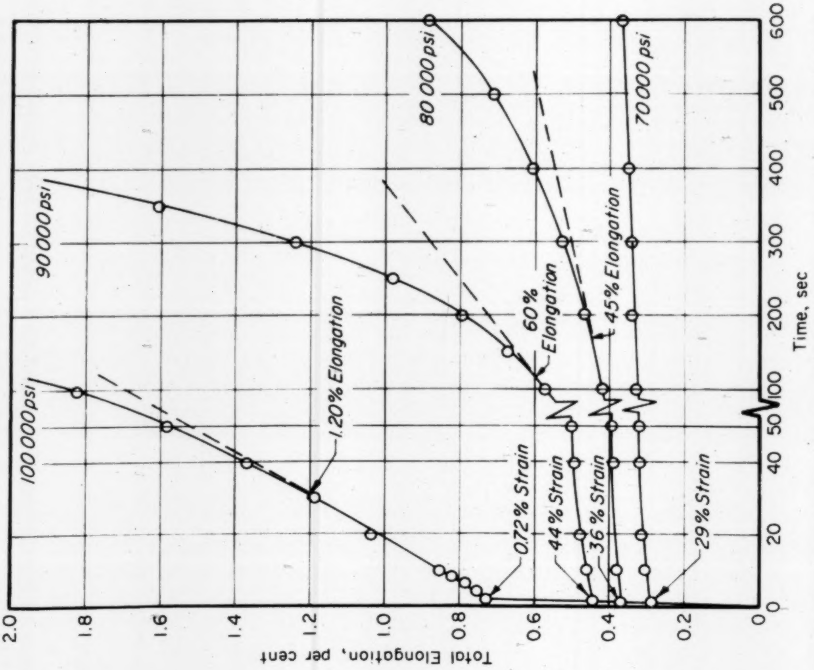


FIG. 9.—Short-Time Tension Creep Deformation of Inconel 718 Alloy Sheet at 1400 F Under Different Constant Loads as Indicated. Mill annealed and aged at 1300 F for 16 hr.

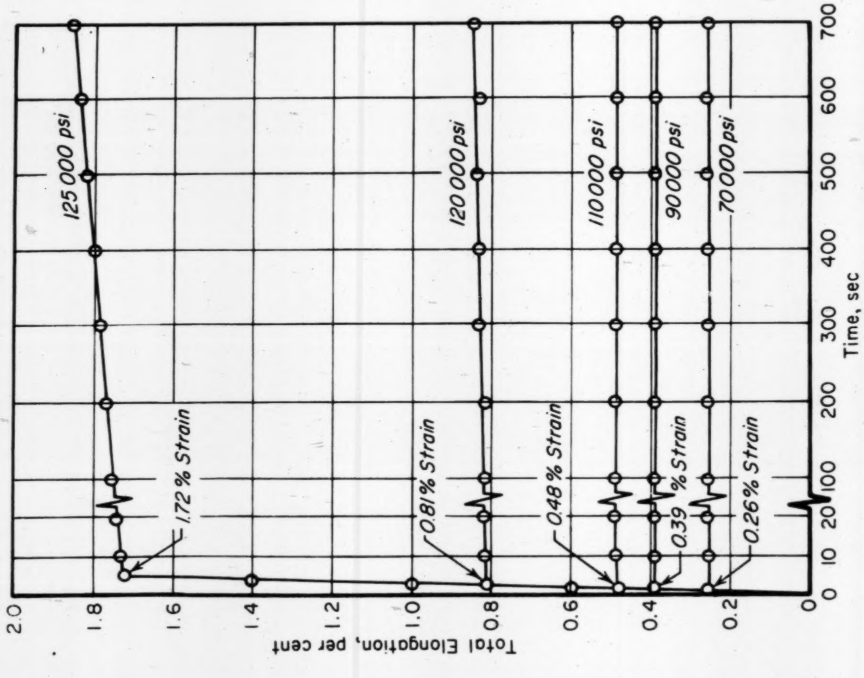
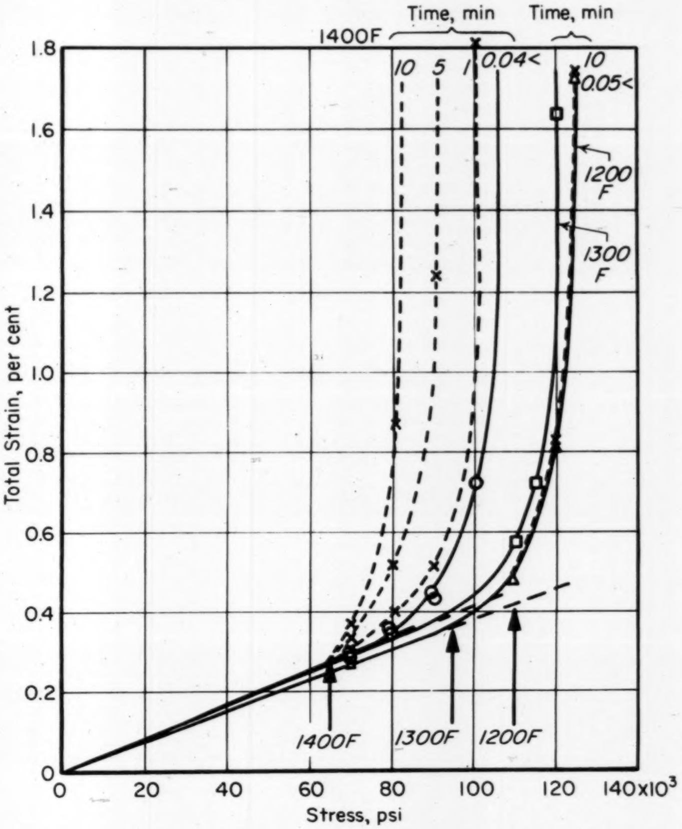


FIG. 8.—Short-Time Tension Creep Deformation of Inconel 718 Alloy Sheet at 1200 F Under Different Constant Loads as Indicated. Mill annealed and aged at 1300 F for 16 hr.

The creep resistance was determined from the very short-time tension curves illustrated in Figs. 8 and 9. These curves were obtained with a dead weight creep machine. The specimen was heated directly by resistance at a rate of 100 F per sec to the desired temperature, held for about 2 min, and load was applied. The temperature control was ±10 F on a 2-in. gage length. The strain was measured with an accuracy of 0.0005 in. The smallest detectable creep thus was approximately 0.02 per cent for the 10-min tests or 0.002 per cent per min.



Solid lines—Strains on loading (ϵ_0) at a recorded rate of 0.21 in. per in. per min.
Dotted lines—Isochronous stress-strain curves as indicated.

Fig. 10.—Short-Time Creep Data Plotted on Stress-Strain Diagrams for Inconel 718 Alloy.

The values of strain on loading, ϵ_0, which were reasonably uniform for duplicate specimens, are plotted directly as the stress-strain diagram (solid lines) in Fig. 10. They appeared to vary linearly with stress up to a certain point, then sharply deviated from linearity due to the strain by plastic gliding. The strain rate recorded during loading was about 0.21 in. per in. per min. The rates higher than this value could not be measured. The change of the loading strains with time for a given temperature are represented by a family of curves, known as isochronous stress-strain curves (dotted lines), in Fig. 10. The stress-strain lines for any strain rate can be derived from these curves by the step-by-step method (12). The use of the isochronous stress-strain curves in design will be considered later.

The creep for short time (1 to 10 min) at a temperature of 1200 F and a stress of say 70,000 psi could not be measured, and the creep curve was represented by a horizontal line (see Fig. 8). The line was, however, slightly concave upward, and a creep rate of 0.001 to 0.002 per cent per hr could be detected by the long-time tests (10 to 100 hr). The short-time detectable creep rate at constant temperature (about 0.002 per cent per min) was observed above a certain level of stress. For a small range of stress slightly above this level, the creep rate remained constant immediately after loading. At higher stresses, creep was accelerated a short time after loading and became catastrophic.

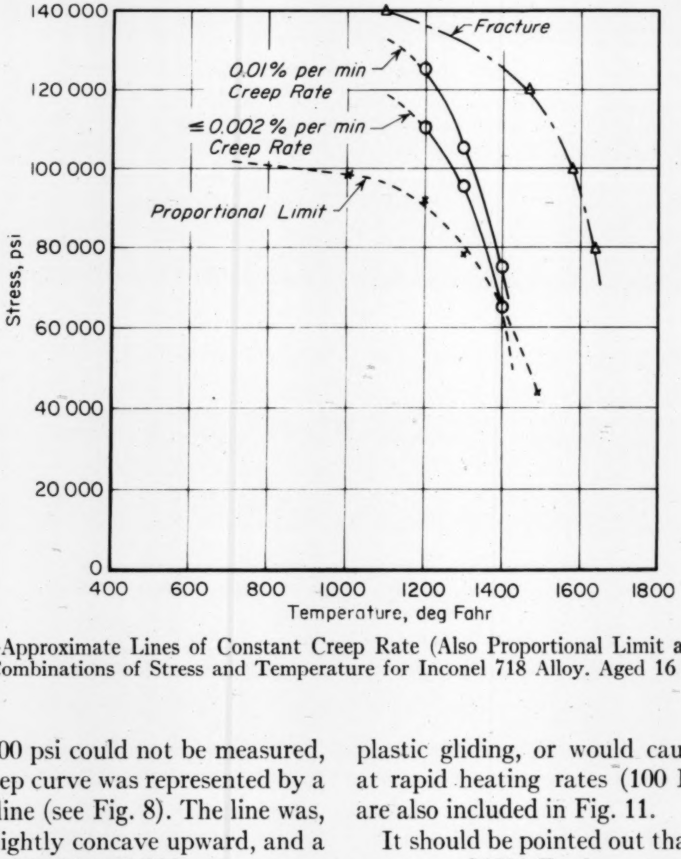


FIG. 11.—Approximate Lines of Constant Creep Rate (Also Proportional Limit and Fracture) at Various Combinations of Stress and Temperature for Inconel 718 Alloy. Aged 16 hr at 1300 F prior to test.

The various combinations of stress and temperature for the detectable creep rate of 0.002 per cent per min and a constant rate of 0.01 per cent per min are shown in Fig. 11. The combinations of stress and temperature which would initiate plastic gliding, or would cause fracture at rapid heating rates (100 F per sec), are also included in Fig. 11.

It should be pointed out that at a temperature of 1200 F, where stress and time-induced hardening occurs, the detectable creep was observed at a relatively high stress, and a small amount of plastic gliding occurred before the creep strain could be observed. The creep curves exhibited a number of steps (about 0.01 to 0.02 per cent strain) associated with the cooperative - slip phenomena (13,14).

Above 1300 F, where thermal recovery and softening by the growth of precipitate become pronounced, the stress producing a detectable creep was comparatively low and plastic gliding did not occur during loading before the creep stress level was reached (see Fig. 10). The creep curves did not exhibit discontinuous yielding. The resistance to strain was rapidly deteriorated, and the creep became catastrophic a short time (300 sec) after loading.

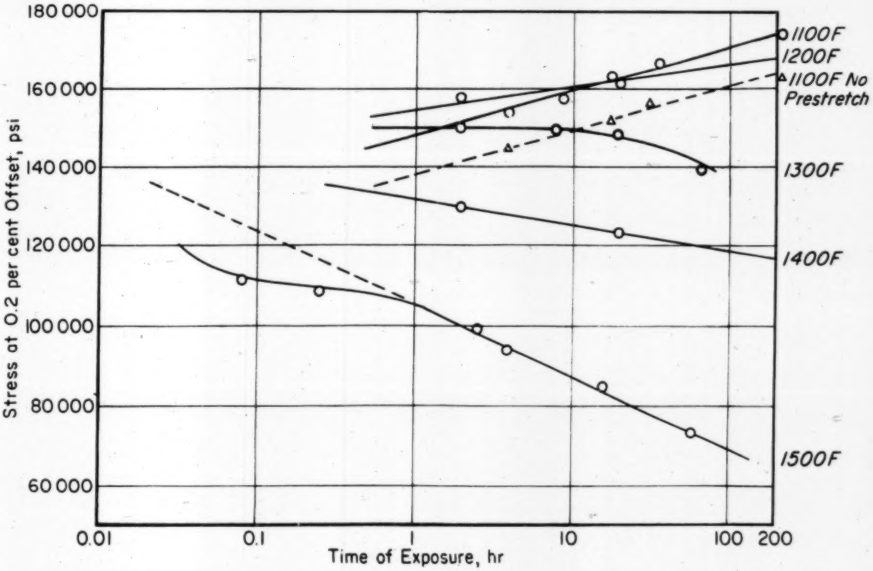


FIG. 12.—Effect of Prestretch and Isothermal Exposure on Yield Stress of Inconel 718.

Specimens aged at 1300 F for 16 hr, prestretched 4 per cent at room temperature, and exposed isothermally at various temperatures as indicated.

It was also noted that when a small load is alternately added and removed at a temperature of 1200 F and a stress of 120,000 psi, the load strains and creep rates after reloading were somewhat smaller than before removal of load. These changes were gradually reduced and became undetectable in two or more successive cycles, depending on the magnitude of load addition and removal. They were apparently related to strain hardening and time-induced precipitation.

Deterioration of Properties:

The resistance of Inconel 718 alloy to plastic gliding and to creep drops sharply at temperatures of 1300 F and above. This drop off in general results from the increase of the thermal vibrations and the diffusion rates of atoms which lead to an accelerated glide and climb of dislocations in the matrix grains (**11**,**13**). These processes also control the stability or the rate of growth of the precipitate particles. The result is that when the load strains and creep are increased, the rate of growth of the precipitate also becomes pronounced and the deterioration of strength is accelerated.

A quantitative evaluation of the effect of the rate-controlling processes on the deterioration of hardness and deformation resistance of Inconel 718 alloy may be made by means of interrupted exposure tests. The results of such tests are shown in Figs. 12 and 13. The interrupted tension tests were conducted by straining the specimens at room temperature for 0.4 per cent, removing the stress and exposing the specimen to different temperatures for various periods of time, and retracing the load-strain curves.

It was noted that the effect of isothermal exposure on the yield strength could be expressed, within certain limits, by an approximate linear logarithmic time relation of the type (**15**,**16**):

$$\sigma = a + m \log t$$

The slope of the straight lines, m, may be interpreted as the "aging index." The temperature dependence of this index was not a simple function. It had a positive value at the temperatures of 1200 F and below, indicating a hardening by time-induced precipitation. It became negative at the temperatures of 1400 F and above, indicating a softening by thermal recovery and growth of the precipitate. At the temperature of 1300 F, it changed from zero to a negative value after an exposure of over 20 hr. The parallelism of the lines for the specimens exposed to a temperature below 1200 F with and without prestretch indicated that the effect of prior small strains on the precipitation hardening was negligible.

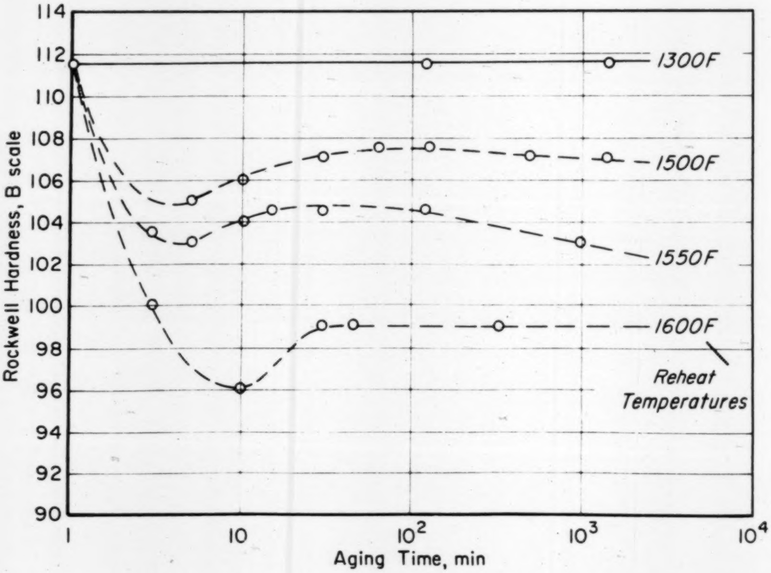


FIG. 13.—Effect of Reheating Temperature and Time on Room Temperature Hardness of Inconel 718 Alloy Aged at 1325 F for 16 hr Before Reheating.

The linear logarithmic time relation for stress broke down at the temperature of 1500 F for very short periods of exposure. This behavior was clearly shown by the hardness curves in Fig. 13. The aged specimen apparently had reverted to the solution annealed condition within 3 to 5 min. This inversion or retrogression phenomenon was similar to the aging minima observed by Mondolfo in aluminum alloys (**17**). It appeared to be associated with the dissolution of some initial precipitate particles near the grain or subgrain boundaries and the coalescence of others at the subgrain or grain boundaries.

Another deformation behavior, which was associated with the stress and time-induced precipitation at the temperatures of 1200 F and below, was the "strain aging maxima." When a prestretched specimen of Inconel 718 alloy was exposed to a temperature between 1000 and 1200 F, its load-strain curve exhibited an upper and a lower peak. The stress required for plastic gliding was increased, and, after an initial plastic strain, the stress began to decrease with increasing strain until a large extension took place. The amount of plastic strain which occurred before the maximum stress was increased, and the total elongation between the upper and lower peaks was decreased with increasing exposure time for a given temperature and prestretch (see Fig. 14).

The authors believe that the strain aging maxima appear to be associated with the precipitation of the gamma prime phase at the sub-boundaries defined by networks of dislocations and the breakdown of the network arrangement. These processes are consistent with the observations of the gamma prime phase at the slip bands and with the lowering of the elongation between the upper and lower peaks with increasing exposure time. They seem to be different from the Cottrell locking of individual dislocations by interstitial atoms, a mechanism proposed for the yield point phenomena in steel and other alloys (13). Similar processes also appeared to be associated with the discontinuous steps observed on the short-time creep curves at 1200 F.

These various mechanical behaviors and precipitation mechanisms are of great importance in the analysis of the time-dependent strains for the structural components in high-speed flight.

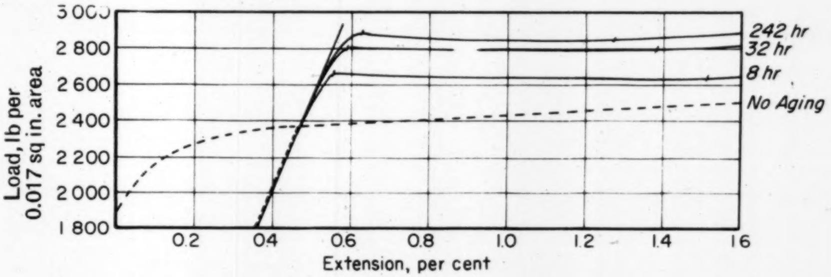


FIG. 14.—Load-Extension Curves for Inconel 718 Alloy Sheet Illustrating Strain Aging Maxima.

Specimens which were stabilized at 1300 F for 16 hr were stretched for 0.4 per cent, unloaded, aged at 1100 F for various periods of time as indicated, and tension tested.

RELATION OF FAILURE OF STRUCTURAL ELEMENTS TO ALLOY PROPERTIES

The failure of the structural elements of a vehicle operating at high temperatures generally occurs by deformation due to excessive stress or to the creep and deterioration of the strength of the material used. The following brief discussion may clarify the relation of these failures to the high-temperature strength characteristics of Inconel 718 alloy.

In general, the structural elements of the multispar wing of a glide vehicle may exhibit four modes of failure by excessive deformation in flight: namely, (1) the onset of permanent buckling of the skin panels, (2) the loss of dynamic stability due to the wing tip deflection, (3) the central panel deflection leading to flutter and excessive drag, and (4) the loss of structural stability resulting from the panel column deflection.

Onset of Permanent Buckling:

The onset of permanent buckling of the skin panels in a multispar wing results in permanent compression wrinkles and necessitates the replacement of the wing or the part affected. The stress required for the onset of permanent buckling, σ_{pb}, depends on the panel thickness and width, the edge constraint, the difference between skin and web temperatures, and the parameter, F_{ty}/E, for the material where F_{ty} is yield strength and E is Young's modulus (**18**). The variation of the values of σ_{pb} during a flight for a lower panel of wing made of Inconel 718 alloy is shown in Fig. 15.

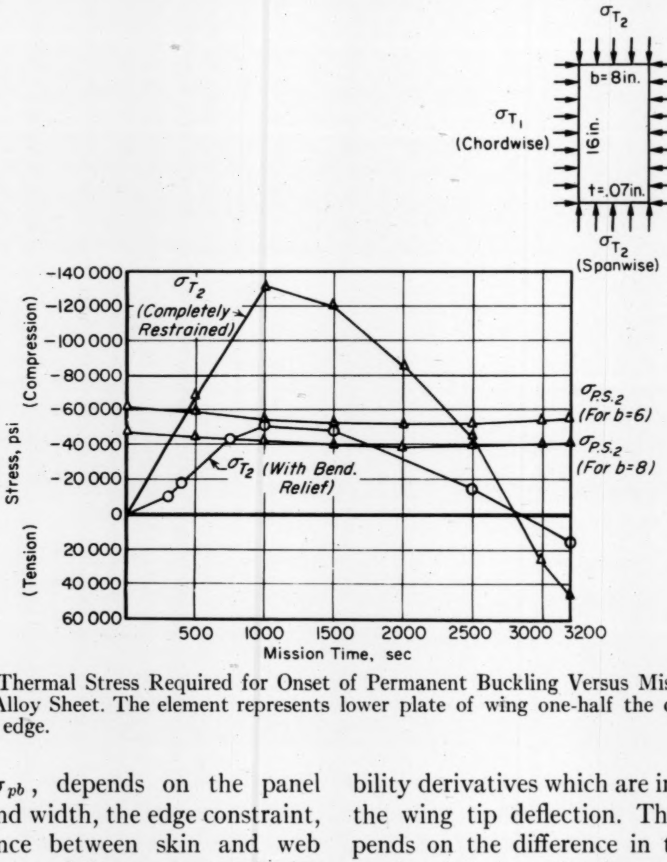


FIG. 15.—Thermal Stress Required for Onset of Permanent Buckling Versus Mission Time for Inconel 718 Alloy Sheet. The element represents lower plate of wing one-half the chord distance from leading edge.

It should be pointed out that the resistance of Inconel 718 alloy to the onset of permanent buckling is high, as long as the temperature does not exceed the critical range of 1200 to 1400 F, where the Young's modulus and the yield strength sharply drop and the latter begins to deteriorate with time.

Loss of Dynamic Stability:

The loss of dynamic stability of a vehicle is due to the changes in the stability derivatives which are influenced by the wing tip deflection. The latter depends on the difference in thermal distortion between the lower and upper spar caps. The lower caps in general are at a considerably higher temperature than the upper caps, as the weight of the vehicle is supported by higher positive pressures on the lower wing surface (**19**, **20**).

The wing tip deflections depend on the

spar depth, the beam length from fuselage intersection to tip, and the effect of temperature on the parameter $E\alpha$ for the lower and upper caps, where E is Young's modulus and α is the thermal coefficient of expansion for the material used. The carpet plot shown in Fig. 16 illustrates the effect of the lower surface temperatures on the wing tip deflections for the structural elements made of Inconel 718 alloy.

It should be pointed out that the effect of temperature on the parameter $E\alpha$ causes recoverable wing tip deflection. During repeated flights, creep and plastic gliding may cause additional nonrecoverable deflections in some elements so that the total accumulated wing deflection may exceed the allowable limits.

The creep strains for Inconel 718 alloy at temperatures below 1300 F and under the loads encountered in the basic flight mission can be assumed additive. When a total strain of 1.2 per cent had thus been accumulated in about 20 flight missions, the wing tip deflection exceeded the allowable limit and induced the loss of dynamic stability of the particular vehicle considered. The planned maneuver (360-deg turn) shown in Fig. 2 also appeared to cause excessive wing tip deflection and loss of dynamic stability.

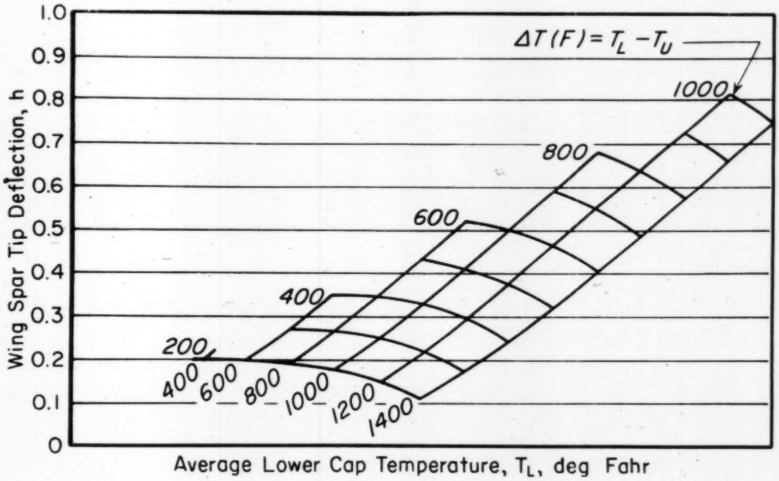


FIG. 16.—Variations of Wing Spar Tip Deflection, h, as a Function of Average Lower Cap Temperature (T_L) and Temperature Difference Between Spar Caps for Inconel 718 Alloy Sheet. Spar length 75 in. and depth 25 in.

Excessive Central Panel Deflection:

The excessive central deflection of the skin panels, which is induced by pressure loading, thermal gradients, and thermal expansion, may cause panel flutter, high drag, and poor vehicle control characteristics. The central panel deflection depends on the panel thickness and width, normal pressure on the panel, normal stress on its edge, and the values of the thermal coefficient of expansion and tangent modulus for the material used (**21**). The deterioration of the tangent modulus with exposure to high temperatures will undoubtedly increase the central panel deflection.

Loss of Structural Stability:

The loss of structural stability results from the excessive panel column deflections. If these deflections exceed certain critical values, the structural member would collapse and the load would increase in the surrounding members. When these cannot sustain the increased load, a progressive failure occurs until the whole wing structure collapses.

The panel column loading per unit length, q, which would induce structural instability is determined by the effective length, L_0, and shape of the column, the radius of gyration of the skin and cap, and the tangent modulus, E_t, of the material used (22). The dependence of the allowable loading parameter, q/L_0, on the panel weight per unit area and temperature is illustrated in Fig. 17 for Inconel 718. It will be noted that the sharp reduction of the tangent modulus at high stress above 1200 F causes a rapid increase in the panel weight necessary to sustain load without collapsing.

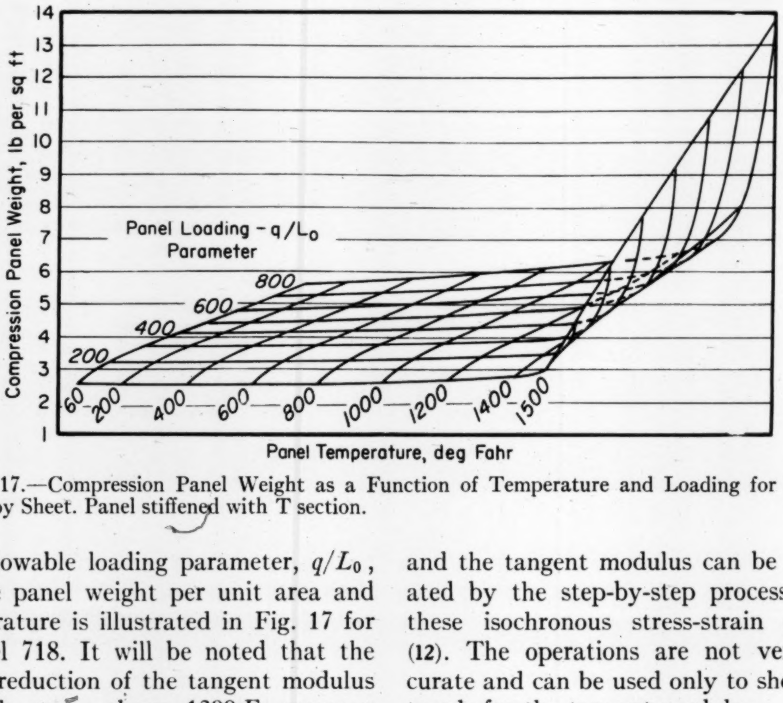


FIG. 17.—Compression Panel Weight as a Function of Temperature and Loading for Inconel 718 Alloy Sheet. Panel stiffened with T section.

The evaluation of the tangent modulus, $E_t = \frac{d\sigma}{d\epsilon}$, appears to be of prime importance for the structural design considerations and analyses of structural failures. A brief comment on this evaluation for Inconel 718 alloy may therefore be in order.

It was noted that the time-dependent portion of the total strain was extremely low at temperatures below 1300 F for Inconel 718 alloy (see Fig. 10). The stress-strain curves for these low temperatures can, therefore, be obtained unambiguously from the short-time tension tests and can be used to determine tangent modulus. At temperatures above 1300 F, the time-dependent strains become pronounced. However, the stress-strain relations at a given temperature can be represented by a family of curves, and the tangent modulus can be evaluated by the step-by-step process from these isochronous stress-strain curves (12). The operations are not very accurate and can be used only to show the trends for the tangent modulus.

SUMMARY

The precipitation hardening mechanism increases the strength of the nickel-chromium alloy Inconel 718 at temperatures up to 1200 F. Above this temperature, however, the precipitate particles grow, and the coherency hardening mechanism begins to break down. These processes create a critical temperature range for the alloy, which is 1200 to 1400 F. Above this temperature range, the strain-induced hardening is eliminated and the resistance to plastic gliding and to creep deteriorate at an extremely rapid rate.

The high-strength characteristics of the precipitation-hardened Inconel 718 make it very attractive for use as a structural material for glide vehicles. The existence of a critical temperature range for the precipitation reactions, however, makes it necessary to evaluate the temperature-stress-time conditions of the flight missions in detail, and to use the alloy only for those structural components that are exposed to temperatures below this critical range of 1200 to 1400 F during the basic flight mission.

The precipitation process is associated with a number of characteristic deformation behaviors such as strain aging maxima, discontinuous steps on the creep curves, serrations on the load-strain curves, variations of load strains and creep rates in successive stress cycling, and retrogression phenomenon. The morphologic features of these phenomena are, however, not very clear. Further investigations with the electron microscope of the mode of precipitation at the sub-boundaries would be invaluable.

A better understanding of the nature of these deformation behaviors and precipitation mechanisms can undoubtedly help to improve the analyses of the time-dependent strains and the accumulated nonrecoverable deflections, and thus the structural design of glide vehicles.

Acknowledgments:

This paper is based on a research conducted at the Douglas Aircraft Co. under Contract AF 33(616)-6680, initiated by Mr. R. F. Klinger, project engineer, Materials Central, Wright Air Development Division of ARDC. The authors are grateful to their co-workers for performing the design analyses and physical tests contributing to the content of the paper, and to The Huntington Alloy Products Division of the International Nickel Co., for supplying alloy samples and some information on alloy behavior.

REFERENCES

(1) J. E. Fischler, J. W. Lobbett, J. W. Stanwood, J. E. Davis, D. Saltman, and N. A. Tiner, "Investigation into More Complete Use of Structural Materials Through a Study of the Stress-Temperature-Time Conditions of a Re-entry Vehicle," WADD Technical Report 60-363, May, 1960.

(2) M. P. Buck, private communication.

(3) A. Taylor and R. W. Floyd, "The Constitution of Nickel-Rich Alloys of the Nickel-Titanium-Aluminum System," *Journal*, Institute of Metals, Vol. 81, p. 25 (1952–1953).

(4) L. O. Brockway and W. C. Bigelow, "The Investigation of Minor Phases of Heat Resistant Alloys by Electron Diffraction and Electron Microscopy," WADC Technical Report 54-589, 1955.

(5) R. Nordheim and N. J. Grant, "Aging Characteristics of Nickel Chromium Alloys Hardened with Titanium and Aluminum," *Journal of Metals*, Am. Inst. Mining and Metallurgical Engrs., Vol. 200, p. 211 (1954).

(6) R. W. Guard and J. H. Westbrook, "Alloying Behavior of Ni_3Al (phase)," General Electric Research Laboratory Report 58-RL-2124, Nov., 1958.

(7) R. F. Decker and J. R. Mihalisin, "Phase Transformations in Nickel-Rich Nickel-Titanium-Aluminum Alloys," International Nickel Co. Research Laboratory Technical Paper No. 276, April, 1959.

(8) R. O. Williams, "Aging of Nickel-Base Aluminum Alloys," General Electric Research Laboratory Report 58-RL-1925, April, 1958.

(9) R. J. Morris, "New Higher-Strength Nickel-Base Alloy," *Metal Progress*, Am. Soc. Metals, Dec., 1959, p. 67.

(10) J. H. Hollomon and J. D. Lubahn, "The Flow of Metals at Elevated Temperatures," *General Electric Review*, Feb., 1947, p. 28, and April, 1947, p. 44.

(11) G. M. Schwab, "Some New Aspects of the Strength of Alloys," *Transactions*, Faraday Society, Vol. 45, p. 385 (1949).

(12) F. R. Shanley, "Weight-Strength Analysis of Aircraft Structures," McGraw-Hill Book Co., New York, N. Y. (1952).

(13) A. H. Cottrell, "Dislocations and Plastic Flow in Crystals," University Press, Oxford (1953).

(14) A. W. McReynolds, "Plastic Deformation Waves in Aluminum," *Transactions*, Am. Soc. Metals, Vol 41, p. 32 (1949).

(15) J. H. Hollomon and L. D. Jaffe, "Time-Temperature Relations in Tempering Steel," *Transactions*, Iron and Steel Division of AIME, Vol. 162, p. 223 (1945).

(16) N. M. Erkun, "The Application of Rate Process Theory to the Heat Treatment of Titanium Alloys," *Transactions*, Am. Soc. Metals, Vol. 51, p. 787 (1959).

(17) L. F. Mondolfo, "Retrogression in Age Hardening," *Transactions*, Metallurgical Society of AIME, Vol. 215, p. 289 (1959).

(18) G. W. Zender and R. A. Pride, "The Combinations of Thermal and Load Stresses for the Onset of Permanent Buckling in Plates," NACA Conference on Aircraft Loads, Structures and Flutter, Langley Field, March, 1957.

(19) J. E. Fischler, "Methods for Rocket Booster Aircraft and Missiles," Douglas Technical Paper No. 781, April, 1959.

(20) P. H. Denke, "A Matric Method of Structural Analysis," *Proceedings*, Second U. S. National Congress of Applied Mechanics, June, 1954.

(21) D. M. W. Lindquist, "Large Deflection Analysis for a Plate Strip Under Combined Pressure and Spanwise Varying Temperature," California Inst. of Technology, Galcit SM 59-12, June, 1959.

(22) W. R. Micks, "A Method of Estimating the Compressive Strength of Optimum Sheet-Stiffener Panels for Arbitrary Material Properties, Skin Thickness, and Stiffener Shapes," *Journal of Aeronautical Sciences*, Vol. 20, p. 705 (1953).

THE TENSILE PROPERTIES OF HARD-DRAWN ALLOY A PHOSPHOR BRONZE ROD

By G. R. Gohn[1] and W. G. Bader[2]

Synopsis

The accepted theory on the mechanism of the cold drawing process indicates that values of tensile strength obtained in tests on full-size rod should be higher than values obtained on subsize specimens machined with their axes coincident with the axis of the rod. However, tests on specimens machined from two lots of hard-drawn alloy A phosphor bronze rod indicate that this is not the case; instead, the tensile strength and yield strength at 0.2 per cent offset increase as the diameter of the subsize specimens decreases. The reverse is true for values of proportional limit and yield strength at 0.01 per cent offset.

In a recent investigation of the fatigue properties of hard-drawn phosphor bronze rod, the data indicated that two lots of material had been interchanged by the machine shop which made the R. R. Moore rotating-beam fatigue specimens. To verify this assumption, some small diameter (0.232-in.) tension specimens were machined from the broken ends of the fatigue specimens. Tests on these specimens showed no mixup in the two lots. However, much to our surprise, the subsize specimens had somewhat higher tensile strengths than were obtained in tests made on the full-size rod stock from which the fatigue specimens were made. To determine the extent of this variation, a series of specimens of different diameters was made from each lot and tested.

Materials Tested

The materials tested consisted of two lots of $\frac{1}{2}$-in. diameter, hard-drawn alloy A (5 per cent tin) phosphor bronze rod made by the Riverside Metal Division, H. K. Porter Co. of Riverside, N. J. One lot was cold drawn from material given a 0.035-mm ready-to-finish anneal, the other from material given a 0.008-mm ready-to-finish anneal. Both lots had the nominal reduction by cold drawing customarily used in the manufacture of hard-drawn rod (approximately 37 per cent reduction in diameter during cold drawing).

Test Methods

The various mechanical properties of the two lots of phosphor bronze rod were determined in accordance with the following applicable ASTM methods:

---

[1] Bell Telephone Laboratories, Inc., New York, N. Y.

[2] Bell Telephone Laboratories, Inc., Murray Hill, N. J.

[3] Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1958 Book of ASTM Standards, Part 3, p. 103.

Tentative Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 57 T), 1958 Book of ASTM Standards, Part 3, p. 39.

Standard Method of Test for Diamond Pyramid Hardness of Metallic Materials (E 92 – 57), 1958 Book of ASTM Standards, Part 3, p. 52.

Test	ASTM Methods[3]
Tension	E 8 – 57 T
Rockwell hardness	E 18 – 57 T
Diamond pyramid hardness	E 92 – 57

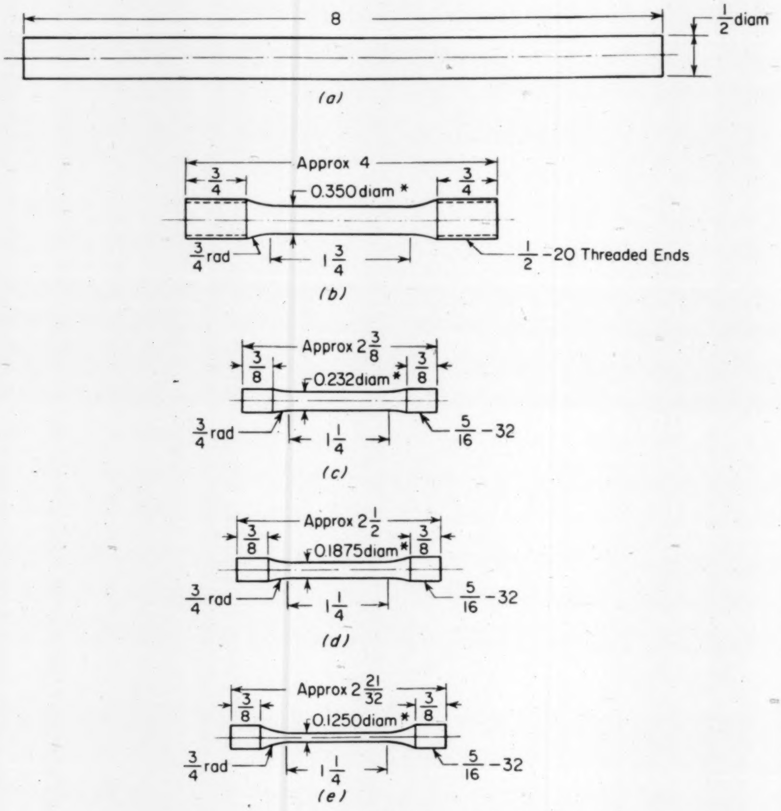

8
1/2 diam
(a)
Approx 4
3/4
3/4
0.350 diam *
3/4 rad
1 3/4
1/2 -20 Threaded Ends
(b)
Approx 2 3/8
3/8
3/8
0.232 diam *
3/4 rad
1 1/4
5/16 -32
(c)
Approx 2 1/2
3/8
3/8
0.1875 diam *
3/4 rad
1 1/4
5/16 -32
(d)
Approx 2 21/32
3/8
3/8
0.1250 diam *
3/4 rad
1 1/4
5/16 -32
(e)

Fig. 1.—Tension Test Specimens. (* ±0.001 in.)

For the tension tests, specimens having the dimensions shown in Fig. 1 were used. The full-size rod specimens were tested without any flattening of the ends. The subsize specimens all had threaded ends. The tension tests were made on a 50,000-lb capacity Baldwin-Emery SR-4 testing machine (model FGT). In determining the elastic properties on the ½-in. diameter specimens, a T1M microformer extensometer with a 2-in. gage length was used in conjunction with an O. S. Peters recorder. On the subsize specimens, the T1M extensometer was replaced by a T2M extensometer having a 1-in. gage length. Elongation values were determined after fracture using a gage length four times the diameter except that the minimum gage length used was 1 in.

The hardness tests were made on polished flats on the ends of rod sections except for the readings reported for a diameter of 0.500 in. which were made on the cylindrical surface. The values reported for the latter are uncorrected for curvature. Rockwell hardness values were taken on the B scale; diamond pyramid hardness values were obtained using a 1.0-kg load.

TEST DATA

The data obtained in this study are recorded in Tables I and II. The former shows the results of tests on the rod cold drawn from material having a 0.035-mm ready-to-finish anneal, the latter for material having a 0.008-mm ready-to-finish anneal. These results are portrayed graphically in Figs. 2 and 3, respectively.

TABLE I.—MECHANICAL PROPERTIES OF ½-IN. DIAMETER HARD-DRAWN ALLOY A PHOSPHOR BRONZE ROD.

Nominal Ready-to-Finish Grain Size, 0.035 mm.

Nominal Diameter of Test Specimen, in.	0.500	0.350	0.232	0.1875	0.1250
Tensile strength, psi	91 700	94 400	96 800	98 700	99 300
Yield strength at 0.2 per cent offset, psi	81 800	82 300	84 200	83 300	86 300
Yield strength at 0.01 per cent offset, psi	65 200	62 000	60 600	58 100	56 200
Proportional limit, psi	59 000	58 400	56 100	52 300	50 500
Modulus of elasticity, psi	15.53×10^6	14.83×10^6	14.90×10^6	14.75×10^6	14.58×10^6
Elongation, per cent in gage length of 4 × *D*	23.1	17.7	13.4[a]	12.9[a]	9.2[a]
Rockwell hardness, B scale	91.7	95.0	96.1	95.0	93.6
Diamond pyramid hardness, 1.0-kg load	217	226	229	215	224

[a] Minimum gage length of 1 in.

TABLE II.—MECHANICAL PROPERTIES OF ½-IN. DIAMETER HARD-DRAWN ALLOY A PHOSPHOR BRONZE ROD.

Nominal Ready-to-Finish Grain Size, 0.008 mm.

Nominal Diameter of Test Specimen, in.	0.500	0.350	0.232	0.1875	0.1250
Tensile strength, psi	94 500	97 200	101 400	102 800	106 900
Yield strength at 0.2 per cent offset, psi	86 600	87 800	89 900	89 900	95 100
Yield strength at 0.01 per cent offset, psi	71 900	69 600	65 900	64 500	62 000
Proportional limit, psi	67 000	62 600	56 200	55 700	53 900
Modulus of elasticity, psi	16.02×10^6	16.38×10^6	16.25×10^6	16.13×10^6	16.63×10^6
Elongation, per cent in gage length of 4 × *D*	24.5	18.9	15.6[a]	11.6[a]	7.6[a]
Rockwell hardness, B scale	94.1	95.0	96.5	96.9	96.7
Diamond pyramid hardness, 1.0-kg load	208	220	217	216	210

[a] Minimum gage length of 1 in.

DISCUSSION OF DATA

The data from the mechanical tests on the two lots of ½-in. diameter hard-drawn alloy A phosphor bronze rod show a similar pattern. In both cases the tensile strength and yield strength at 0.2 per cent offset increase with decreasing diameter of the test specimens. In other words, as the supposedly more severely cold drawn outer surface is machined away, the residuum appears to have higher strength. In a similar but somewhat less pronounced manner the Rockwell and the diamond pyramid hardness values are less on the outer surface than on the intermediate diameters.

The proportional limit, yield strength at 0.01 per cent offset, and the elongation at fracture in a 4 × D gage length show the reverse effect, namely a decrease in value with decreasing diameter of the test specimen. The decrease in the elongation values is accentuated for the two smallest diameters by the use of a minimum gage length of 1 in. No marked pattern is shown by the modulus values. For the coarser grain size rod the modulus values increase slightly from edge to center, for the finer grain size material the opposite appears to be true.

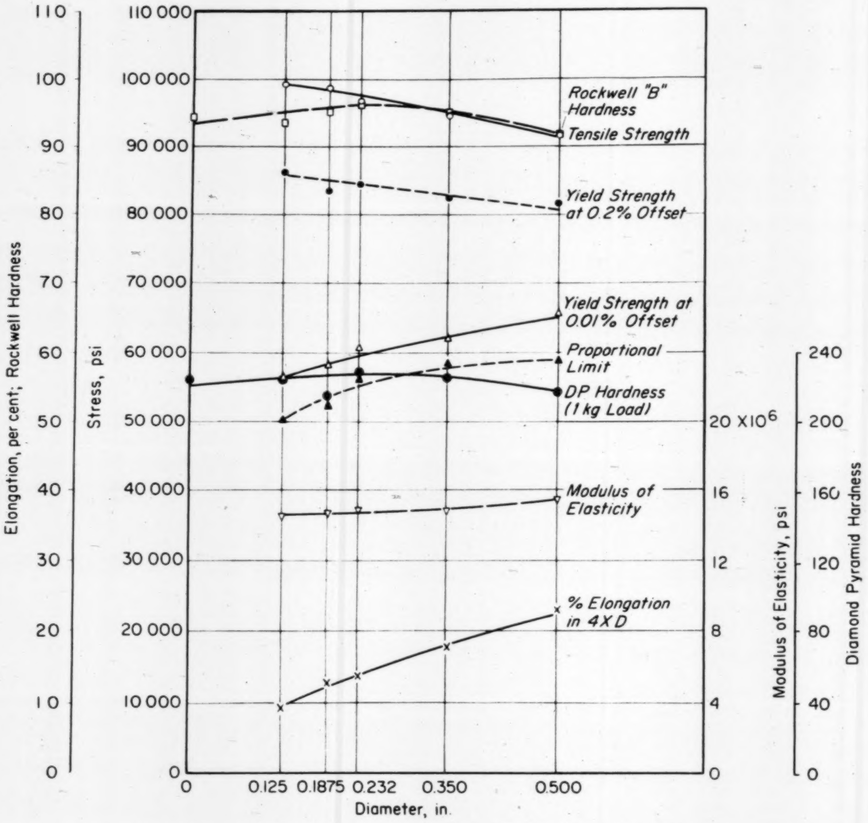


FIG. 2.—Mechanical Properties versus Diameter of Test Bar.

Hard-drawn 5 per cent tin phosphor bronze rod.
Nominal diameter, 0.500 in.
Nominal grain size, 0.035 mm.

The values for tensile strength and the 0.2 per cent offset yield strength observed in these studies on hard-drawn phosphor bronze rod are not consistent with our usual interpretation of the cold drawing process. Cold drawing is a strain hardening process and results in greater strength and hardness in cold drawn material such as the phosphor bronze rod studied. Since it is normally assumed that the strain hardening decreases from surface to center of the rod, there should be a decrease in strength in the residuum as the outer portion of the rod is machined off (as in the case of the subsize specimens). Inasmuch as the opposite effect was observed, this would lead one to believe that our interpretation of the cold drawing process is not in accordance with the facts. Whether this condition prevails on all hard drawn rod or is peculiar to the alloy, diameter of rod, and per cent reduction by cold rolling used in the manufacture of the two lots tested cannot be determined from this study. Conversations with two suppliers of phosphor bronze rod indicate that these findings are wholly unexpected and that further study of the cold drawing process is desirable for a better understanding of the nature of the process.

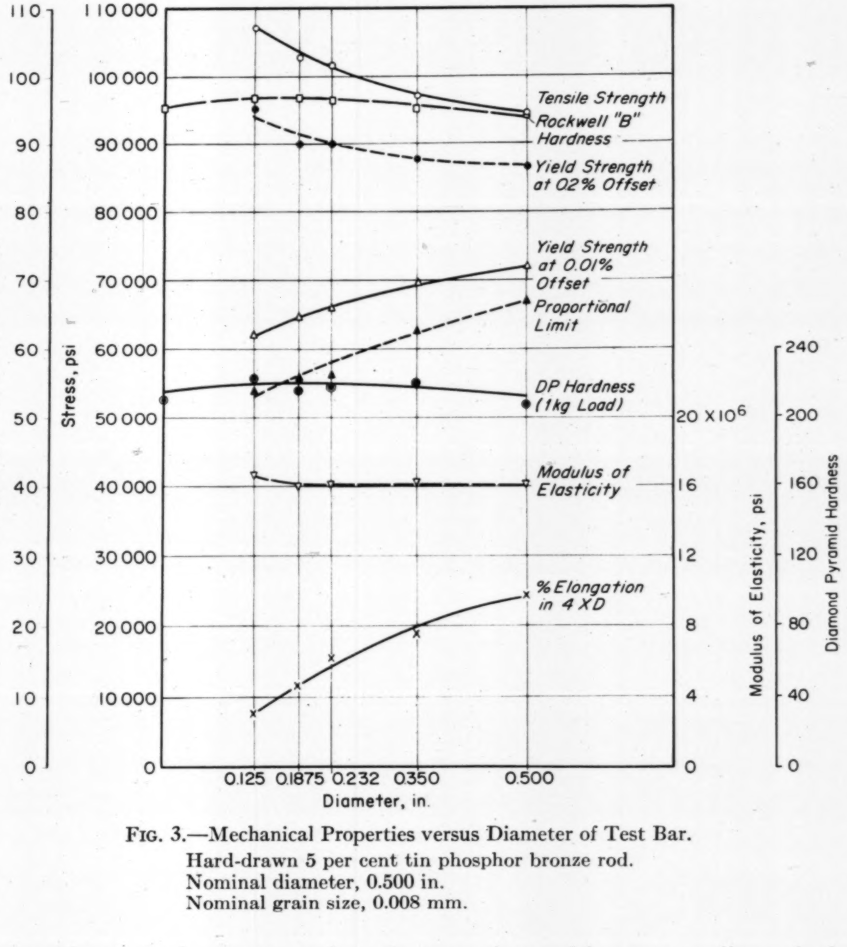


FIG. 3.—Mechanical Properties versus Diameter of Test Bar.
Hard-drawn 5 per cent tin phosphor bronze rod.
Nominal diameter, 0.500 in.
Nominal grain size, 0.008 mm.

Furthermore, these data would indicate that the usual practice of estimating other mechanical properties from the tensile strength or hardness may lead to erroneous conclusions. Definitely it cannot be assumed that the proportional limit or yield strength at 0.01 per cent offset can be estimated as a fixed percentage of the tensile strength. In fact, the data show an inverse relationship. Neither can the practice of reporting the fatigue strength in terms of a percentage of the tensile strength be condoned because the tensile strength varies with specimen diameter. If the diameters of the fatigue and tension specimens differ, then this procedure results in a comparison of material in two different conditions. Testing procedure dictates that, wherever possible, the tension test should be made on full-size rod (see ASTM Method E 8 - 57 T, section 5(*a*)). On this basis tensile values for the 0.500-in. diameter specimens would normally be used in comparing tensile properties and fatigue. But fatigue tests are usually made on rotating beam specimens having a diameter of 0.300 in. Such a specimen would have a tensile strength some 4 to 5 per cent higher than that of the full-size rod. (If a smaller diameter fatigue specimen was used the disparity would be even greater.) Hence all fatigue data in the literature for drawn rod which are based upon a percentage of the tensile strengths are questionable unless additional data are given on the size of the original rod and the diameter of the test specimen used.

CONCLUSIONS

Subsize specimens machined from hard-drawn rod will show mechanical properties which are substantially different from those of the full-size rod. In the case of the tensile strength and the 0.2 per cent offset yield strength, the values increase with decreasing specimen diameter. The reverse is true in the case of values for the proportional limit, 0.01 per cent offset yield strength, and elongation at fracture for gage lengths equal to 4 × D. No general pattern was observed in the case of the elastic modulus or hardness values.

THE ROLE OF REFRACTORY METALS IN SUPERALLOYS*

By Earl R. Parker[1]

Synopsis

An understanding of the basic mechanism of creep is an essential prerequisite for understanding how the elements molybdenum, tungsten, vanadium, and columbium act to reduce creep and prolong the life of superalloys. The physical processes controlling creep, namely, diffusion and dislocation climb, are therefore discussed briefly. The effects of various factors that affect high-temperature strength, such as grain size, elements in solution, the presence of a second phase, and heat treatment, are discussed in terms of their influence on the physical processes that control creep at elevated temperatures. Data are presented which show that both molybdenum and tungsten are important solid solution strengtheners, but that molybdenum is superior to tungsten. Vanadium and columbium are particularly effective strengtheners because they form stable carbides that interfere with the movement of dislocations. Complex alloys are also shown to be stronger than simple ones, and the reasons for this are presented. Also discussed are the reasons why the most effective strengthening is produced by a combination of solid solution strengtheners and carbide formers. The paper is meant to be interpretive in the sense that the behavior of commercial high-temperature alloys is explained in terms of the modern physical theory of creep.

Alloys used for high-strength applications in the temperature range of 1200 to about 1800 F are herein referred to as "superalloys." Such materials are oxidation resistant and can be fabricated in large quantities, although with more difficulty than ordinary stainless steels. They consist of complex solid solutions (iron-nickel-chromium, nickel-chromium, or nickel-cobalt-chromium) strengthened by a dispersion of relatively stable carbides or particles of an intermetallic compound. Molybdenum, tungsten, vanadium, and columbium are particularly effective strengtheners, contributing to creep and stress-rupture resistance both by solution and dispersion hardening (as carbides). Another effective combination of elements sometimes found in superalloys is aluminum and titanium which react to form a stable intermetallic compound.

The development of high-temperature creep resistant materials has been an empirical process involving the making and testing of hundreds of alloy combinations, with the effort finally culminating in the selection and further refinement of the best of the alloys found in the more general search. This work was guided by such general established facts as: (1) nickel and chromium are essential for oxidation resistance; (2) cobalt in solid solution improves high-temperature strength; and (3) refractory metals are needed to form stable compounds and also contribute to solid solution strengthening. Basic principles, however, could not be applied to guide the alloy development because of the primitive state of knowledge about flow and fracture processes. Theory has finally evolved to a fairly satisfactory state, but it reached maturity about a decade after the period of alloy development. A restudy of superalloy characteristics, interpreted in terms of modern theory of flow and fracture, may prove helpful to those working with such materials, and might even suggest research leading to improved alloys. It is the purpose of this paper to discuss the creep and stress-rupture behavior of the superalloys in terms of modern theory.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Professor of Metallurgy, Department of Mineral Technology, University of California, Berkeley, Calif.

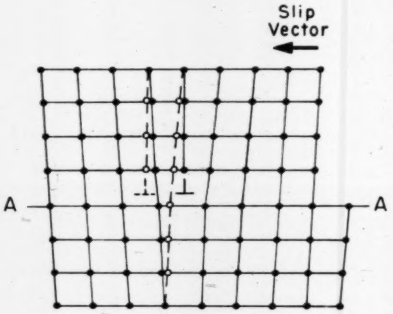


FIG. 1.—Sketch Illustrating Nature of Edge Dislocation in a Simple Lattice. Dotted lines show new position of dislocation after translation on plane *A-A* caused by application of a shear stress.

THEORETICAL CONSIDERATIONS

Dislocation Mechanism of Plastic Deformation:

High-temperature tests and their evaluation are well understood (**1**,**2**),[2] but why metals creep and why they break with intergranular fractures are questions that cannot be simply answered. It is well known that plastic flow, both at ordinary and at high temperatures, occurs when close-packed planes of atoms in metal crystals slide past one another. However, the planes of atoms do not glide over each other like cards in a deck because the stress required to move an entire plane of atoms as a unit would be higher by a factor of 100 or more than the stress needed to make real metals flow plastically (**3**). Taylor (**4**) and Orowan (**5**) proposed in 1934 that the weakness of real crystals was due to a crystalline defect called the "edge dislocation" or "line dislocation." Taylor showed theoretically how this kind of defect could account for strain hardening as well as for the observed low yield strength of metals. An elementary form of dislocation is illustrated by Fig. 1. Here the defect is shown as an imperfect square grid with the top half having one more vertical row of atoms than the bottom half. In a three-dimensional model of the simple cubic lattice there would be a large number of planes identical to the one shown, but spaced evenly in front and in back of it. The three-dimensional defect consists of a missing sheet of atoms in the crystal. A line through the lattice along the top of the missing sheet of atoms (perpendicular to the plane of the drawing) would coincide with the region of maximum lattice disturbance. Dislocations can thus be represented by such a line; it is for this reason that this type of dislocation is called a "line" or "edge" dislocation. Above or below the dislocation the lattice spacing gradually returns to its normal value. The atoms are squeezed together above the line and spread apart below it. The shear stresses are a maximum on the plane (*AA* in Fig. 1) perpendicular to the missing sheet of atoms, and it is on this plane that slip occurs. At the dislocation line, the shear stresses are extremely high and a small additional shear stress, provided by an external load, can cause the dislocation to move through the lattice in the direction of the applied stress; such motion is indicated by the dotted lines in the figure. When dislocations reach free surfaces, they provide a slip effect of one interatomic distance. Visible slip lines are produced when hundreds of dislocations on the same or nearby planes escape at the surface. It has long been known that plastic flow is concentrated in "slip bands," but why this is so was not understood until 1950 when Frank and Read (6) showed that many dislocations could be generated on a single slip plane by a simple lattice defect called the "Frank-Read dislocation source." Experiments in recent years have proved that the Frank-Read sources do exist and that they do function as originally postulated (3,7). Thus the reason for the observed large slip offsets (many hundreds of atomic spacing) has been satisfactorily explained.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The next question in need of an answer was "Why does a source stop functioning after hundreds of dislocations have been generated?" This question is actually the same as "What causes strain hardening?" because plastic flow on a slip plane stops when a source ceases to function. Additional flow cannot, then, occur unless the stress is increased sufficiently to activate new sources. An understanding of this phenomenon has been provided by an analysis of the stress fields that surround dislocations. Since some regions of the lattice exist in a state of tension while others are in compression, there may be attraction or repulsion forces acting between two dislocations, depending upon their relative positions in the lattice. For example, two identical dislocations lying on the same slip plane repel each other, whereas two on nearby parallel planes are mutually attracted and tend to align themselves one above the other. Both of these interactions are extremely important. The repulsion interaction is responsible for strain hardening, whereas the attractive action has a major influence on creep behavior. In recent years it has become increasingly clear that strain hardening is caused by dislocations interacting with each other and with barriers, such as precipitated particles and grain boundaries, which impede their progress through the metal. Concepts of flow and fracture at low temperatures have recently been summarized (8,9) and so only phenomena related to high-temperature creep will be discussed herein.

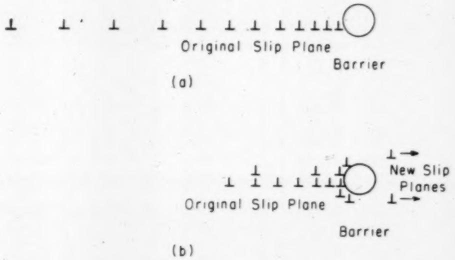


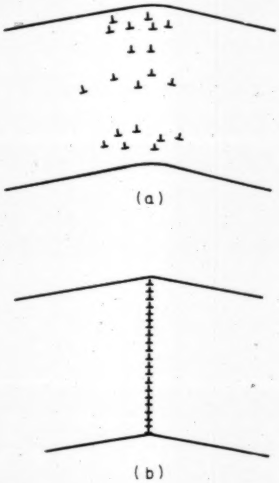
FIG. 2.—Sketch Illustrating (*a*) How Barrier Can Cause Dislocations to Pile-up on Slip Plane and (*b*) How Climb Permits Dislocations to Detour Around Barriers.

Cause of High-Temperature Creep:

An understanding of the basic mechanism of creep is essential for an understanding of high-temperature behavior of metals. It is now well established that plastic flow at both low and high temperature is mainly due to the movement of dislocations through the lattice. Furthermore, it is well known that dislocation movement is confined to planes of high atomic density, the "slip planes," because these are the only ones on which dislocations can move at ordinary levels of stress. (Movement on other planes is possible, but higher stresses would be required.) Thus all dislocations generated by a single source normally travel on the slip plane of their origin until they reach a free surface, or until they encounter a barrier strong enough to stop their march through the lattice. A barrier causes dislocations to pile up, one behind the other, with the last one in the pile being so close to the source that it becomes impossible for the source to send out another dislocation. A dislocation pile-up is shown schematically in Fig. 2(*a*). At low temperatures, such piled-up dislocations are permanently stuck in the lattice and the metal remains indefinitely in the strain hardened state. At elevated temperatures, similar pile-ups occur, but such arrays change slowly with time because a new phenomenon, known as "dislocation climb," occurs. At temperatures above about 0.4 of the absolute temperature melting point, dislocations acquire a new degree of freedom. They can move through the lattice in a direction perpendicular to the slip plane, an event that cannot occur at low temperatures. This movement is very slow, however, compared to the speed with which dislocations can move on a slip plane. Climb is a slow process because it depends upon the diffusion of atoms (or vacancies), and the thermal migration of atoms is a sluggish process. Diffusion occurs at high temperatures because the atoms are thermally activated, vibrating through substantial amplitudes about their nominal lattice positions, and because the lattice contains vacant lattice sites as well as dislocations. The vacant lattice sites, or vacancies, as they are commonly called, wander from site to site in the lattice because of the thermal vibration. Both the rate at which they migrate and the fraction of vacancies in the lattice increase exponentially with temperature. When a vacancy wanders near a dislocation, it tends to unite with it because the free energy of a vacancy attached to a dislocation is generally less than that of a vacancy in a lattice site. In the two-dimensional lattice shown in Fig. 1, the capture of a vacancy by the dislocation would have the effect of moving the dislocation upward so that it would reside on a new slip plane. A continuation of this process would cause the dislocation to move many atom distances in a direction perpendicular to the slip plane. By a similar process, the deposition of atoms on a dislocation could cause downward climb. In a three-dimensional lattice, climb would require the deposition on the dislocation of rows of vacancies or atoms. The climb process thus requires the transport of many atoms by the process of self-diffusion; diffusion, therefore, governs the rate of dislocation climb, and this, in turn, governs the rate of creep. For every dislocation that is moved out of a pile-up by the climb process, another dislocation can be generated by the source to replenish the pile. Climb, therefore, makes continued generation, and hence creep, possible. Eventually a dislocation may climb far enough so that it can detour around the obstacle that blocked its movement on the original slip plane. When this happens, the dislocation can then move forward on a parallel slip plane and thus contribute to the continuing process of plastic flow. The climb phenomenon is illustrated by the sketch shown in Fig. 2(*b*).

According to theory, the rate-controlling process in creep is self-diffusion. The activation energy for diffusion and creep should, therefore, be identical. This has been checked by many experiments and found to be so (**10**). The coincidence of activation energies, coupled with even more direct evidence, has demonstrated conclusively that dislocations do climb, just as theory dictates they should. It is now generally agreed that the rate of high-temperature creep is controlled by self-diffusion.

In the temperature range in which dislocations are free to climb, they frequently arrange themselves one above the other because this arrangement corresponds to a minimum energy configuration. Figure 3 shows how dislocations piled up on slip planes tend to rearrange themselves at high temperatures. Boundaries of this kind are called by various names, such as dislocation boundaries, small angle boundaries, and subboundaries. X-ray evidence has shown that within a single grain of polycrystalline metal, a number of such boundaries form during creep. These boundaries form a complex stable network within the grains that resists the passage of moving dislocation. Their formation is thus associated with an increase in strength of the metal (**11**,**12**).



(*a*) Before heating.

(*b*) After heating to a temperature high enough for rapid dislocation climb.

FIG. 3.—Sketch Illustrating Change Produced by Heating a Plastically Bent Crystal.

The bending over of a creep curve of a metallurgically stable material is due to the development of a substructure during the creep test; increasing amounts of substructure result in decreasing rates of creep. This has been demonstrated by tests in which controlled amounts of substructure were introduced prior to the initiation of the creep tests (**12**). Results illustrating the effect are shown in Fig. 4. The substructure was introduced by prestraining the creep specimens at room temperature to the amounts indicated, and then heating them to 800 C prior to creep testing to permit the dislocations to rearrange themselves into boundaries. The amount of substructure was checked by X-ray diffraction techniques and was found to increase nearly linearly with the amount of prestrain. The initial creep

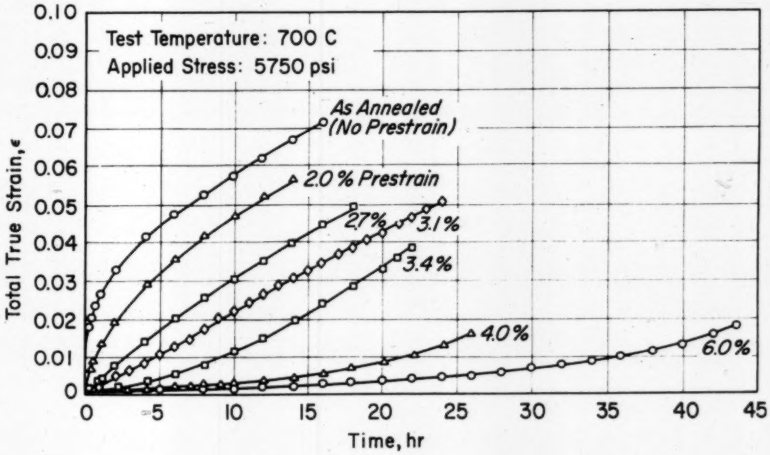


FIG. 4.—Effect of Substructure Introduced by Prestraining Indicated Amounts and Annealing at 1470 F on Shape of Creep Curve at 1290 F for High-Purity Nickel (**12**).

rate decreased with increasing amounts of substructure (as indicated by per cent prestrain), and the shape of the creep curve was strongly dependent upon the substructure state of the material. As Fig. 4 shows, it is possible to change the shape of the creep curve of a metal from concave downward to a straight line, or even to make it concave upward (a condition that arises when more than the equilibrium amount of substructure is introduced prior to the initiation of the creep test and some of it disappears during creep). The "steady-state" of creep in metallurgically stable metals is evidently the rate that corresponds to that of the equilibrium substructure state. Note that in Fig. 4, the creep rate tends to approach a constant steady value independent of the initial substructure state of the material. It is interesting to note that the so-called "hot-cold work," or "warm-work," used to improve the high-temperature properties of certain gas turbine alloys is the kind of treatment that introduces a substructure in the alloys prior to testing or use. Plastic working at intermediate temperatures introduces dislocations that form subboundaries during and immediately after the working operation; this happens because the working temperature is high enough to permit rapid dislocation climb.

Effect of Grain Boundaries on Creep:

In polycrystalline materials, the creep process is complicated by the presence of grain boundaries. While it is true that such boundaries interfere with moving dislocations, and thus contribute to the strength, it has also been known for many years that adjacent grains can move past one another by shear on or near the boundary separating them. It has long been thought that grain boundary sliding is a major cause of creep of polycrystalline metals, particularly at high temperatures. It is well known that fine-grained materials creep faster at elevated temperatures than do the same materials with a coarse-grain structure, whereas at low temperatures fine-grained materials are invariably stronger. The excessive creep of fine-grained materials has been attributed to grain boundary sliding, which contributes proportionately more to the plastic flow when the grain size is small. Some recent work, however, has shed considerable doubt upon the validity of this explanation of the grain size effect. Investigations carried out by several groups (**13–15**) have shown that grain boundary sliding does occur, but that it probably contributes less than 10 per cent to the total creep strain; hence, grain boundary shear cannot account for the grain size effect in creep. Grain boundaries are known to be effective barriers to moving dislocations at high temperatures as well as at low temperatures. Therefore, fine-grained material should normally be stronger in creep than coarse-grained material. This has not been found to be so in most of the experimental work reported in the literature; consequently, it appears that some other factor is exerting a controlling influence on creep. Another important fact is that the activation energy for creep is independent of grain size. Thus it may be concluded that volume self-diffusion (and dislocation climb) control creep regardless of the grain size, and that no new processes are involved.

The main barriers to dislocation motion in single phase materials (generally used to investigate grain size effects) are grain boundaries. Dislocations piled up against such boundaries must climb if creep is to occur, and lattice vacancies must be available to make climb possible. Where do the vacancies come from? The answer to this question is the key to understanding the grain size effect, and it also facilitates the understanding of how alloy additions of refractory metals act to reduce creep rates. Vacancies are not generally created spontaneously within a volume of perfect crystal, but they form most easily at external surfaces or at grain boundaries where an atom, agitated excessively by thermal vibrations of the lattice, can most easily move out of a lattice position (leaving a vacancy). Surface atoms can move onto an external surface, and atoms near grain boundaries can move into a void in a boundary. External surfaces are generally not too important in polycrystalline materials because the area of grain boundaries is generally far greater than the area of the free surface. Furthermore, external surfaces are generally covered with an oxide layer that inhibits the free motion of surface atoms. The important surfaces are, therefore, the internal or grain boundary interfaces, and it is these that must be considered.

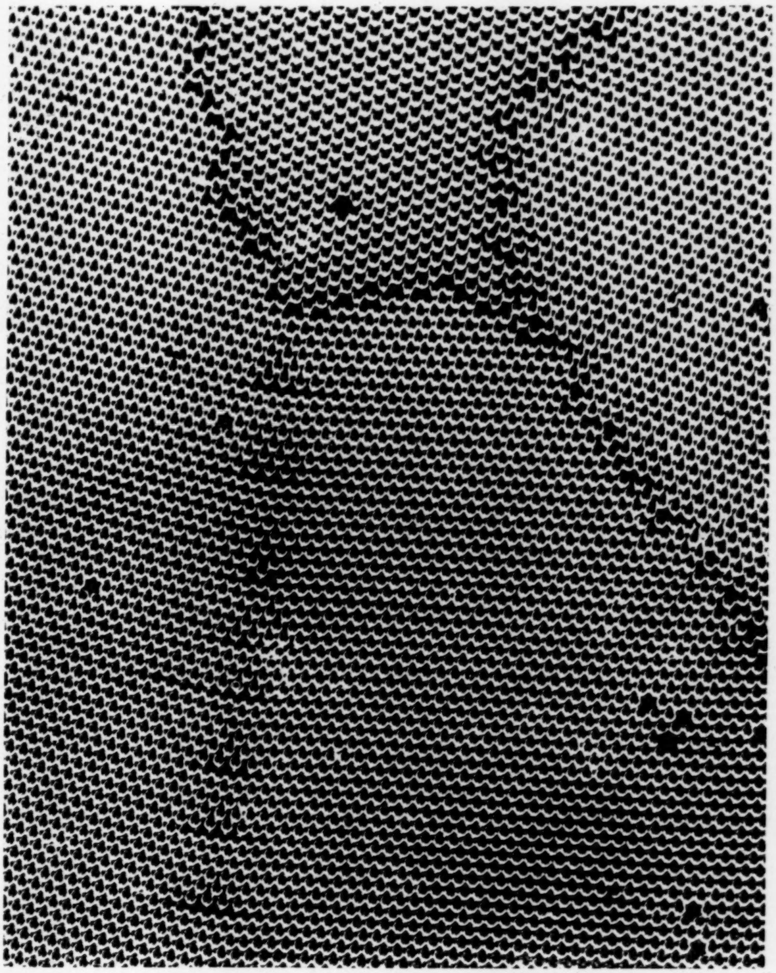
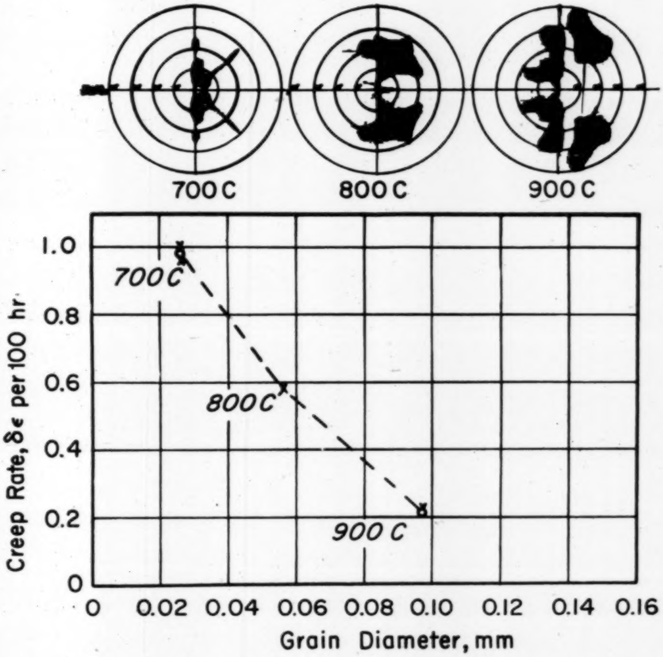


FIG. 5.—Soap Bubble Model of Grain Boundaries Illustrating Variation in Grain Boundary Structure with Various Orientations of Adjacent Grains (after C. S. Smith (**23**)).

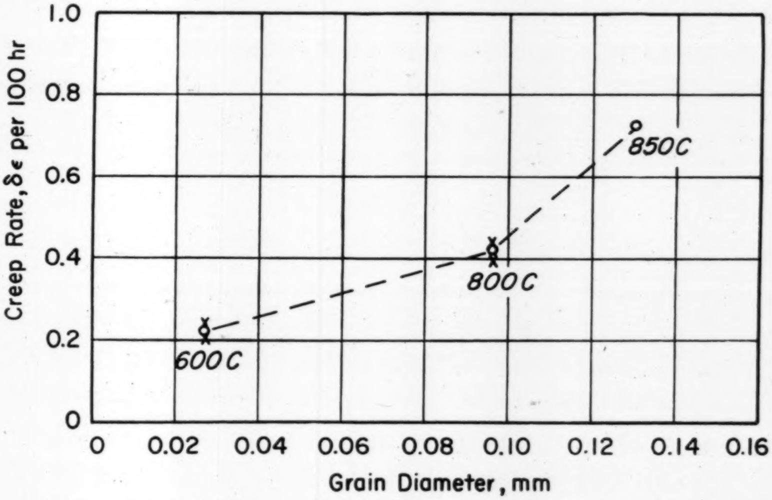
The ability of grain boundaries to provide vacancies constitutes the most important contribution of such boundaries to creep. Recognition of this fact makes it possible to understand seemingly anomalous and inconsistent results. First of all, it must be recognized that the ability of a grain boundary to provide vacancies depends upon the nature of the boundary —that is, upon the relative orientations of the adjacent grains. When neighboring crystals differ only slightly in alignment, there are relatively very few places where atoms from the lattice can find room to move into the boundary. Hence, lattice vacancies would be created infrequently in the grains adjoining small angle boundaries. When adjacent grains are badly misaligned, many places exist where atoms can move into the boundary, and in this case the rate of vacancy generation is relatively high. (The photograph shown in Fig. 5 is helpful in visualizing how the structure of a grain boundary depends upon the relative orientations of the adjacent grains.) The availability of vacancies has a direct bearing upon how fast dislocations can climb, and so it is to be expected that dislocations piled up at small angle boundaries, which are poor providers of vacancies, will climb slower than those adjacent to complex boundaries. Furthermore, complex boundaries can readjust themselves quickly after losing a vacancy because of the very high mobility of grain boundary atoms.

The conclusion from this study is that grain size, as such, is not necessarily the most important factor, but that the nature of the grain boundaries must also be considered when grain size effects are investigated. This leads to an analysis of the procedures generally employed in investigating the effects of grain size on creep. The usual practice is to anneal a cold-worked material at a low temperature to produce a small grain size; medium-sized grains are produced by heating the same material to an intermediate temperature, and large grains are obtained through the use of high annealing temperatures. It is generally assumed that heating to various temperatures changes only the grain size. In reality, however, two variables are involved, and they affect plastic flow in different ways. Enlarging the grain size, contrary to popular belief, reduces the strength at high temperatures just as it does at low temperatures, because the barriers—the grain boundaries—are spaced farther apart and thus dislocations are freer to move over longer distances than are those in their smaller grained counterparts. The anomalous fact, however, is that large grained material, prepared by heating specimens of the same material to a high temperature, creeps slower than fine-grained specimens. What could cause this grain size effect? (Experiments have shown that grain boundary sliding cannot account quantitatively for this behavior.) The answer must be that the nature of the grain boundaries is different in the coarse and fine grained material. How can annealing effect a change in the nature of the boundaries? A study of grain growth characteristics sheds some light on this problem.

Fine-grained material, produced by annealing cold-worked metal at a low temperature, generally has randomly oriented grains. Many grain boundaries in such material are high-energy interfaces that serve as generous sources of vacancies. The same metal, when heated to a higher temperature, tends to retain fewer of the large-angle, high-energy boundaries, because such boundaries are known to disappear preferentially during grain growth (16). Thus, the grain boundaries that remain are the smaller-angle lower-energy ones which are poor providers of vacancies. The dislocation climb process should, therefore, be slower in the coarse-grained material because of the



(*a*) Grain sizes produced by heating material to a series of increasingly higher temperatures. The temperature shown by each point is the annealing temperature used to produce the grain size shown on the horizontal scale. The pole figures above the graph show the degree and kind of preferred orientation existing in the specimens with various grain sizes.



(*b*) Results obtained with randomly oriented grains in all specimens. Randomness obtained by varying working and annealing history so that grain orientations remained random.

FIG. 6.—Creep Rate at a Constant Stress Versus Grain Size for High-Purity Copper Tested at 750 F.

lower rate at which vacancies can emerge from the lower energy boundaries.

Some experiments were performed to check this conclusion in which specimens of copper were prepared by a variety of working and annealing treatments. Pole figures were made to determine the effects of the treatments on the relative orientations of the grains in the polycrystalline aggregates. Randomly oriented specimens were produced with a variety of grain sizes by varying the thermal-mechanical history of the material. The creep rates of these specimens were determined as were those of specimens having the same range of grain sizes, but produced by the conventional manner of heating a fine-grained material to successively higher temperatures to produce larger grains. The results are reproduced in Fig. 6. Figure 6(*a*) shows the behavior normally found with material heated to progressively higher temperatures to increase the grain size. The usual relationship was found—the larger the crystals, the lower the creep rate. Figure 6(*b*) shows the grain size effect in the same material, subjected to the same stress and temperature, but in this case the thermal-mechanical history was altered so that truly random orientation was retained for all grain sizes. In this series *only the grain size was changed*. The results therefore reflect the true grain size effect, namely, that the creep rate increases with increasing grain size when only the grain size is altered.

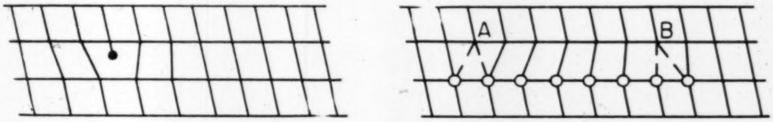
The results shown in Fig. 4 further substantiate the foregoing interpretation of the grain size (and grain boundary) effect in creep. The small angle subboundaries introduced by straining and recovering are very poor vacancy sources, but they are fairly good barriers to moving dislocations. The nature of the subgrain boundaries did not change with increasing amounts of prestrain, but the subgrain size became smaller and the creep rate diminished with subgrain size.

Influence of Alloying Elements and Crystal Structure:

Recognition of the fact that grain boundaries supply the vacancies required for dislocation climb is helpful in understanding creep and stress-rupture behavior. It has long been known, for example, that only a few ounces of silver per ton of copper have a remarkable strengthening effect upon the creep and stress-rupture properties of copper (**17**). The reason for the marked influence of the trace of silver has not been clear, but it now seems evident that the silver atoms, which differ in size from the copper atoms by about 15 per cent, effectively fill the grain boundary voids and thus reduce the rate that a given boundary can contribute vacancies to the lattice. Similarly, an element like molybdenum can be expected to fill the grain boundary voids in iron or the superalloys, and thus reduce creep.

Difference in atomic size is only one important factor. Certain atoms are more mobile than others at creep temperatures, and this affects the rate at which boundaries can readjust themselves after a local loss of vacancies. For example, the element phosphorus has a large effect on the strength of iron at low temperatures, but a small effect at high temperatures. The same atomic concentration of an element like molybdenum has a smaller effect at low temperatures but a much greater effect on the high-temperature strength. The difference at low temperatures, where both atoms are "frozen" in fixed atomic positions, can be attributed largely to their differences in atomic size and valence. At high temperatures, however, the phosphorus atoms move rapidly about and thus cannot retard the readjustment that is required in a grain boundary due to the loss of a vacancy. Molybdenum atoms, on the other hand, are even less mobile than are atoms of iron, nickel, and cobalt at temperatures in the neighborhood of 1800 F, and hence they form effective deterrents to grain boundary readjustments.

It is a well-known fact that the strongest high-temperature alloys (such as the superalloys) have close-packed crystal structures (that is, face-centered cubic or hexagonal close packed). The reason for their superiority has become clear in recent years. Dislocations in body-centered cubic crystals have a simple structure, similar to that portrayed in Fig. 1. In the close-packed structures, however, the dislocations assume a more complicated shape wherein the lattice disturbance is spread out in the lattice instead of being concentrated; this is illustrated schematically in Fig. 7. Such a dislocation is called an "extended" dislocation (3,7). Because the lattice disturbance is spread throughout a much greater area on the slip plane, the maximum local distortion is reduced. More important for the understanding of creep, however, is the fact that the attraction of an extended dislocation for a vacancy is much weaker than that of an ordinary dislocation. Because of their more complex structure, extended dislocations climb slower than the normal type and so creep proceeds at a slower rate in metals having close-packed structures.



(a) (b)

FIG. 7.—Sketch Illustrating Nature of (*a*) Unextended Dislocation and (*b*) Extended Dislocation.

Hexagonal metals tend to creep slower than cubic metals for still another reason. Such materials are inherently more resistant to plastic flow because the limited number of slip systems (usually only three and all of these on one plane) makes it difficult for strain adjustments to occur in adjacent grains. Thus it is not surprising to find that many of the strongest high-temperature alloys have hexagonal crystal structures.

Effect of Second Phase Particles:

Strong nonplastic particles dispersed throughout crystals retard creep because they constitute effective barriers to moving dislocations. At low temperatures where fine particles do not coalesce and dislocations cannot climb, the finer the dispersion of a given quantity of a second phase, the stronger the alloy, because fine dispersions provide the maximum interference to moving dislocations. At high temperatures, maximum strength is not associated with the finest dispersion. There are two reasons why this is so. One is that dislocations can climb around very tiny particles easier than around larger ones; the second reason is that fine dispersions are not stable at creep temperatures. The smallest particles dissolve in the matrix and the larger ones accumulate material and grow in size. When a small particle dissolves, the dislocations that it retarded temporarily are freed so that they can continue to move through the lattice to cause creep. At high temperatures, therefore, there will be an optimum particle size for the precipitate in a given alloy and this optimum will vary with temperature, stress, and time of testing or use. There is, of course, the further complication that the solubility of many second phase particles increases with temperature, and so the effect of a dispersed phase may drop off precipitously in a rather narrow temperature range.

The idea of using particles of a second phase that are substantially insoluble in the matrix below the melting point has been rather thoroughly investigated. Materials of this kind for high-temperature use have been made by powder metallurgy techniques involving mixtures of a metal and a refractory material. Among the most successful of these has been the tungsten carbide - cobalt mixture used to make sintered carbide cutting tools. Some degree of success has been obtained with other and more dilute mixtures, but in almost all cases the "cermets," as they are called, have been found to be too brittle to use in applications such as gas turbine buckets.

COMMENTS ABOUT COMMERCIAL ALLOYS

A brief discussion of the role played by alloying elements in the superalloys, in terms of the dislocation picture of creep, may be helpful in understanding why these materials behave as they do. A review of the work by Freeman and coworkers (**18,19**), who determined the effects produced by 62 modifications of the base compositions of a low carbon N-155 alloy (0.15 carbon, 1.7 manganese, 0.5 silicon, 20 chromium, 20 nickel, 20 cobalt, 3 molybdenum, 2 tungsten, 1 columbium, 0.12 nitrogen, balance iron).

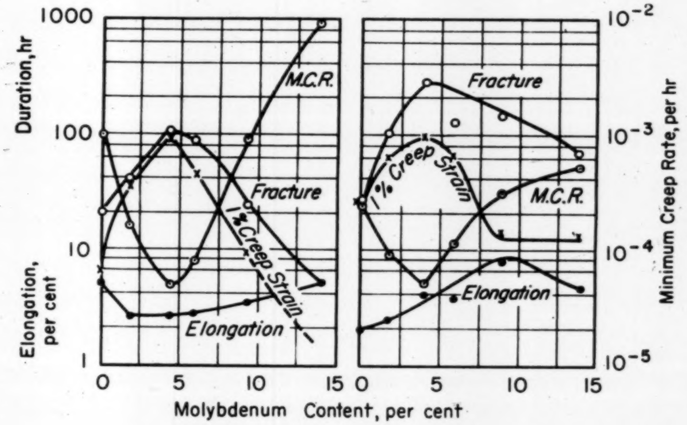
All specimens were solution treated at 2200 F and aged at 1400 F. In rupture tests at 1500 F, they found that the carbon could be increased to about 0.40 per cent without significantly changing the strength, but the elongation at failure was found to be much lower for the higher carbon alloys. The strength was less when the carbon content was below 0.15 per cent. Extensive carbide precipitation occurs in the grain boundaries in alloys containing larger amounts of carbon. Such particles anchor grain boundaries so that lateral movement, an effective means of readjusting grain boundaries from which vacancies have been removed, cannot occur as readily as it can in carbide-free boundaries. Further readjustment of atoms along the boundaries is retarded when carbides are present.

Since the rate at which boundaries can supply vacancies depends upon how rapidly they can readjust themselves to repair the disturbance caused by the loss of vacancies, it is to be expected that the rate of creep would be lowered by the presence of grain boundary carbides. With the life at a given stress being substantially unchanged by increasing the carbon content, it is to be expected that the strain at fracture would be lowered.

Freeman found that the manganese content had to be maintained at or above that of the base composition, or the elongation at fracture decreased. This effect can also be attributed to a grain boundary impurity effect. High nickel and cobalt alloys are notably sensitive to small traces of sulfur, which forms grain boundary sulfides unless sufficient manganese is present to combine with the sulfur to form insoluble manganese sulfide particles that do not concentrate in the grain boundaries.

A decrease to 10 per cent of either nickel or cobalt had no significant effect upon the properties of the alloy, whereas any reduction in the amounts of chromium, molybdenum, tungsten, or columbium below the levels in the base composition caused a decrease in strength. No benefit was derived from increases of chromium, molybdenum, or tungsten, and larger additions of columbium were found to be detrimental. An alloy with 8 per cent molybdenum plus tungsten, but containing no columbium, was found to be just as strong as the base composition but it had less ductility. The alloy had a larger grain size than normal and the grain boundaries contained precipitated carbides. Both factors act to reduce ductility. Columbium was found to be particularly effective as a grain refiner. The stable carbide that it forms acts favorably in two ways: (1) the insoluble carbide particles nucleate many grains during recrystallization and restrict grain growth thereafter, and (2) stable carbide particles do not collect in grain boundaries.

Harris and colleagues (**20**) conducted a similar investigation on an alloy of 20 per cent nickel, 20 per cent chromium, 20 per cent cobalt, 0.5 per cent carbon, various amounts of molybdenum, tungsten, columbium, and vanadium, with the balance being iron. The alloying elements were varied singly, as well as simultaneously, to determine the optimum creep and stress rupture properties. The optimum creep (and stress rupture) properties for the molybdenum series occurred at 4 per cent, as shown in Fig. 8. The rupture strengths of tungsten, columbium, and vanadium alloy series exhibited similar optima at about 15, 12, and 5 per cent respectively. The most significant result of this investigation was the discovery that the effect of combined additions of the refractory elements was greater than the sum of the contributions of the individual elements. In the combined alloy series, the total content was set at 9 per cent. It was found that the alloys containing only one of any of the four elements were the weakest. In almost every case, stronger alloys were found with binary combinations; ternary alloys were strongest, and there was an indication that properly balanced quaternary alloys might be superior. The better properties of the more complex alloys can reasonably be attributed to the fact that the grain boundaries are more effectively plugged by mixtures of solute atoms having different sizes. Furthermore, columbium and vanadium form stable carbides that inhibit grain growth and restrict grain boundary carbide precipitation; ductility is thereby enhanced.



(*a*) (*b*)

FIG. 8.—Effect of Molybdenum Content on Properties of a Base Alloy of 20 per cent Nickel, 20 per cent Cobalt, 20 per cent Chromium, 0.5 per cent Carbon at (*a*) 27,000 psi and 1380 F, (*b*) 18,000 psi and 1470 F (**20**).

The effectiveness of molybdenum and tungsten as solid solution strengtheners is illustrated in Fig. 9, taken from the work of Nisbet and Hibbard (**21**). These investigators determined the properties of over 200 variations of iron-cobalt-nickel-chromium-base alloys. Among these were the series shown in Fig. 9, wherein the molybdenum and tungsten contents were varied in a regular manner so that the effect of solid solution strengthening could be evaluated. It was found that both elements had substantially the same effect when compared on an equal atomic concentration basis. However, more molybdenum than tungsten could be dissolved in the alloy without the formation of a second phase. The marked strengthening induced by the presence of a second phase is clearly illustrated by this work. As soon as the second phase appeared in the alloy, the rate of increase in strength with increasing alloy addition changed to a much higher value. Particles of a second phase, however, particularly when present in the grain boundaries, tend to lower the ductility at fracture. Thus the increase in strength due to second phase particles is generally offset by a decrease in ductility. It is worthy of note that the

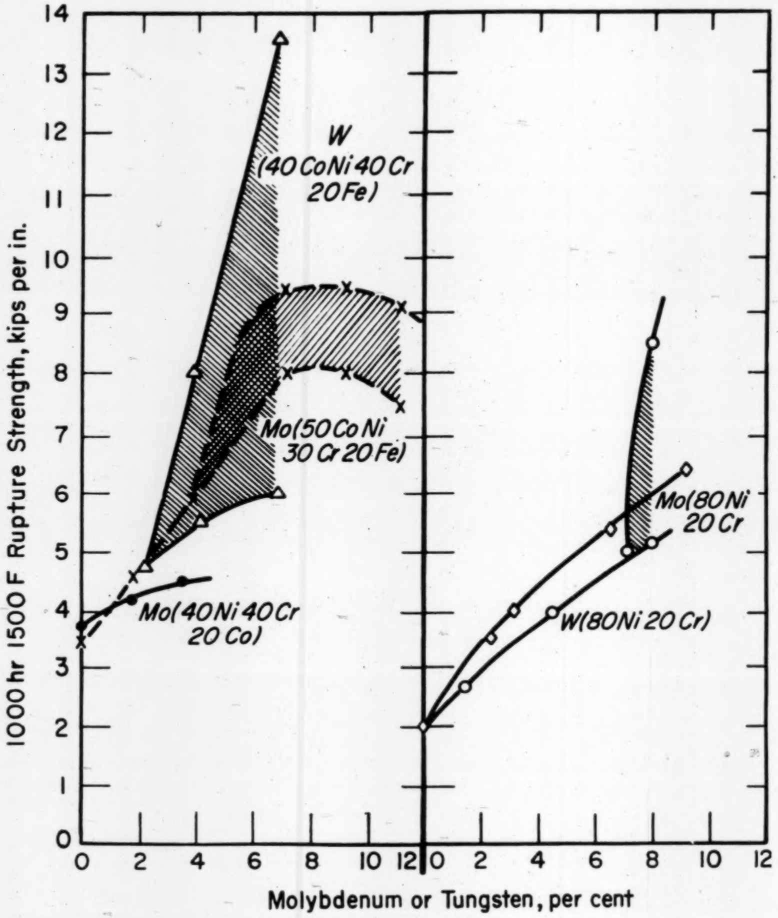


FIG. 9.—Effect of Molybdenum and Tungsten Additions on the 1000-hr Rupture Strength at 1500 F for Several Base Alloy Compositions (**21**).

strength can be increased by a factor of 3 by solution hardening, even at temperatures as high as 1500 F.

COMMENTS ABOUT MANUFACTURING PRACTICE

The beneficial effect produced by the mechanical working of superalloys at temperatures in the neighborhood of 1300 F can be readily explained. This "hot-cold working," or "warm working" as it is commonly called, introduces a substructure in the material that is responsible for the increased strength produced by this kind of working. Frey and co-workers (22) made a systematic investigation of the effect of mechanical working at various temperatures on the creep resistance of low carbon N-155 alloy. Specimens were reduced in cross-section by amounts varying from 5 to 40 per cent at temperatures ranging from room temperature to 2200 F. The materials were air cooled after straining and subsequently annealed for times ranging from 1 to 1000 hr at several temperatures between 1200 and 1800 F. In materials reduced 15 per cent, the grain outlines became progressively clearer as the working temperature was raised; above 1800 F, a small amount of grain boundary precipitation was detected. Microstructures of pieces reduced 15 per cent or less did not differ substantially from that of the starting material, except that recrystallization occurred in bars rolled at 2200 F; no recrystallization was evident in the metal rolled below 2050 F. Recrystallization occurred at lower temperatures in specimens given greater reductions.

Creep tests were conducted at 50,000 psi and 1200 F; a minimum creep rate was found for reductions of 25 per cent. For working temperatures below 1400 F, the effect of a given amount of strain was found to be independent of the rolling temperature. For higher rolling temperatures, the 1200 F creep strength decreased rapidly with increasing rolling temperature. The creep rate was also found to increase with longer annealing times for all annealing temperatures; for a given time of anneal, the creep rate increased with increasing annealing temperature.

As has been previously discussed, subboundaries introduced by mechanical working and heating reduce the amount of strain during the earlier stages of creep. Furthermore, alloying elements causing solution hardening are effective largely because they tend to concentrate in distorted regions of the lattice such as subboundaries and grain boundaries. The subboundaries collect "atmospheres" of foreign atoms which tend to make them more resistant to the penetration of moving dislocations. Elements such as molybdenum and tungsten, which are notably unaffected by temperature in the range where the superalloys are useful, constitute the best kind of solid solution alloying additions. They tend to contribute to the strengthening by pinning dislocation boundaries and by reducing the rate that vacancies can be supplied to the lattice by the grain boundaries.

CONCLUSIONS

1. Creep is controlled by self-diffusion.

2. The basic process responsible for high-temperature creep is dislocation climb.

3. The vacancies required for dislocation climb must be acquired by the dislocations from the lattice.

4. The rate at which dislocations can acquire vacancies is determined by two things: one is the self-diffusion characteristics of the lattice, and the other is the rate at which vacancies can be supplied to the lattice by the source.

5. The temperature dependence of the creep rate is controlled by the self-diffusion process, that is, by the length of

time that it takes for vacancies to diffuse from the source to piled-up dislocations. Hence, the activation energy for creep (that is, the temperature dependence parameter) is identical with that of self-diffusion.

6. The rate of creep at a given temperature depends not only upon how fast vacancies can diffuse through the lattice, but also upon the rate at which vacancies can be transferred to the lattice from the *source*.

7. The main sources of vacancies are the grain boundaries.

8. Grain boundaries vary in their ability to act as vacancy sources. Large-angle high-energy boundaries can provide vacancies at a rapid rate, whereas small-angle low-energy boundaries are poor providers.

9. Two factors are involved in the relation between grain size and creep, the grain diameter and the nature of the grain boundaries.

10. Contrary to the generally accepted belief, smaller grain sizes are associated with lower creep rates and higher strengths at elevated temperatures, provided that comparisons are made on material in which only the grain size, *and not the nature of the grain boundaries*, is varied.

11. In randomly oriented material, the creep rate increases with increasing grain size.

12. Grain size effect investigations in which a range of grain sizes is obtained by heating a material to successively higher temperatures do not reveal the true role played by grain size, because two variables are changed simultaneously by this procedure—the grain size is altered, and the nature of the grain boundaries is changed. The larger-angle higher-energy boundaries tend to disappear preferentially with increasing annealing temperatures. The change in the nature of the grain boundaries often has a more profound effect upon the creep rate than does the change in the grain size.

13. Small-angle boundaries, such as those introduced within the grains by plastic deformation and a "recovery" anneal, are effective in reducing the creep rate at a given stress and temperature. Such boundaries reduce creep mainly because they are poor providers of the vacancies required for dislocation climb.

14. Solid solution elements tend to concentrate in grain boundaries (and in subboundaries) because atoms that are larger or smaller than those of the host material can find less crowded quarters in the relatively disorganized grain boundary material.

15. Solution hardening at elevated temperatures is due to the alloying elements concentrated in the grain boundaries, which tend to fill up the grain boundary voids and thus to reduce the effectiveness of the boundaries as vacancy sources.

16. To be effective as a solution strengthener at elevated temperatures, the alloying element must be less mobile at the creep temperature than are the atoms of the host material. Thus only the refractory metals, such as molybdenum and tungsten, can act as efficient solution strengtheners in the superalloys.

17. Stable particles of a carbide or intermetallic compound effectively improve the creep properties in several ways. They act as barriers to dislocations moving within the grains; they restrict grain boundary migration and the readjustment of atoms within the grain boundaries necessary to repair the local change brought about by the loss of a vacancy from the boundary. Such particles also act to restrict grain growth, and thus they contribute to ductility because finer-grained materials are generally more ductile than coarse-grained ones.

Acknowledgments:

The author wishes to express his appreciation for the assistance provided in the preparation of this paper to Mrs. Rosa Nissen and Mr. R. E. Peterson, who made the pole figures and creep tests on the copper series discussed herein. Special thanks is hereby accorded Dr. J. Z. Briggs of the Climax Molybdenum Co. since it was at her suggestion that this paper was prepared.

REFERENCES

(1) G. V. Smith, "Properties of Metals at Elevated Temperatures," McGraw-Hill Book Co., New York, N. Y. (1950).

(2) A. H. Sully, "Metallic Creep and Creep Resistant Alloys," Interscience Publishers, New York, N. Y. (1949).

(3) A. Cottrell, "Dislocations and Plastic Flow in Crystals," Clarendon Press, Oxford, England (1953).

(4) G. I. Taylor, "The Mechanism of Plastic Deformation of Crystals," *Proceedings*, Royal Society (London), Vol. A145, p. 362 (1934).

(5) E. Orowan, "Plasticity of Crystals, IV. Further Foundation for the Dynamic Plasticity Law," *Zeitschrift für Physik*, Vol. 89, pp. 605, 614, 634 (1934).

(6) F. C. Frank and W. T. Read, "Multiplication Processes for Slow Moving Dislocations," *Physical Review*, Vol. 79, p. 722 (1950).

(7) T. Read, "Dislocations in Crystals," McGraw-Hill Book Co., New York, N. Y. (1953).

(8) E. R. Parker, "Modern Concepts of Flow and Fracture," *Transactions*, Am. Soc. Metals, Vol. 50, p. 52 (1958).

(9) B. L. Averbach, D. K. Felbeck, G. T. Hahn and D. A. Thomas (Editors), "Fracture," Technology Press and Wiley, New York, N. Y. (1959).

(10) J. E. Dorn, "Some Fundamental Experiments on High Temperature Creep," *Journal of Mechanics and Physics of Solids*, Vol. 3, p. 85 (1954).

(11) J. Washburn, "Effect of Structure of Dislocation Boundaries on Yield Strength," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 203, p. 675 (1955).

(12) T. Hazlett and R. D. Hansen, "Influence of Substructure on the Shape of the Creep Curve," *Transactions*, Am. Soc. Metals, Vol. 47, p. 50 (1955).

(13) D. McLean, "Grain Boundary Slip During Creep of Aluminum," *Journal*, Inst. of Metals, Vol. 81, p. 293 (1952).

(14) H. C. Chang and N. J. Grant, "Inhomogeneity in Creep Deformation of Coarse Grained High Purity Aluminum," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 197, p. 1175 (1953).

(15) B. Fazan, O. D. Sherby, and J. E. Dorn, "Some Observations on Grain Boundary Shearing During Creep," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 200, p. 919 (1954).

(16) P. A. Beck, "Annealing of Cold Worked Metals," *Advances in Metal Physics*, Vol. 3, p. 245 (1954).

(17) E. R. Parker, "The Effect of Impurities on Some High Temperature Properties of Copper," *Transactions*, Am. Soc. Metals, Vol. 29, p. 269 (1941).

(18) J. W. Freeman, J. F. Ewing, and A. E. White, "Influence of Chemical Composition on Rupture Test Properties at 1500°F of Forged Chromium-Cobalt-Nickel-Iron Base Alloys," *NACA TN 2745*, July 1952.

(19) E. E. Reynolds, J. W. Freeman, and A. E. White, "Influence of Chemical Composition on the Rupture Properties at 1200 F of Wrought Cr-Ni-Co-Fe-Mo-W-Cb Alloys," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 194, p. 946 (1952).

(20) G. T. Harris and H. C. Child, "Development of a High-Temperature Alloy for Gas Turbine Rotor Blades," *Iron and Steel Institute, Special Report No. 43*, p. 67, July 1952.

(21) J. D. Nisbet and W. R. Hibbard, Jr., "A Rationalization of Measured High Temperature Properties of Fe-Cr-Co-Ni Alloys," *Transactions*, Am. Inst. Mining, Metallurgical and Petroleum Engrs., Vol. 197, p. 1149 (1953).

(22) D. N. Frey, J. W. Freeman, and A. E. White, "Fundamental Effects of Cold Working on Creep Properties of Low Carbon N-155 Alloy," *NACA TN 2472*, Oct. 1951.

(23) C. S. Smith, "A Decade of Metallurgical Science," *Metal Progress*, Vol. 58, Oct. 1950, p. 479.

DISCUSSION

MR. H. C. CROSS.[1]—This has a very practical aspect too, I think. Let us take a gas turbine blade first. If you have a material which you have found by experience to have a useful life of 5000 hr and if you should run a blade for 4000 hr, is there anything in this fundamental work which you have been discussing which would lead us to believe that some heat treatment might be applied to restore the material to its original strength so that instead of running it for just 1000 hr more, you might run it say 3000 or 4000 hr more and get 8000 hr life out of it instead of 5000?

MR. E. R. PARKER (*author*).—I think that is within the realm of possibility, but the problem involved is the nucleation and growth of the grain boundary cracks. I have been talking about creep prior to the formation of intergranular cracks. Less is known about intergranular cracking than about creep, so I cannot answer your question specifically. Perhaps when we learn more about crack nucleation, it will become possible to extend the useful life of high-temperature materials.

---

[1] Metallurgist and Assistant Technical Coordination Director, Battelle Memorial Inst., Columbus, Ohio.

RELAXATION CHARACTERISTICS OF INCONEL AT ELEVATED TEMPERATURES*

By C. R. Kennedy[1] and D. A. Douglas[1]

Synopsis

A stress relaxation machine has been developed capable of providing very precise results throughout the useful temperature range of common structural metals. Among the advantages claimed for this machine are: dynamic response, sensitive control, and low cost.

It is demonstrated that with the use of a single specimen and relatively short testing times, a complete set of stress-strain rate values can be obtained at any desired temperature. The reliability of the testing device and the analytical model is shown in the comparison of values predicted from relaxation tests with those obtained in constant-stress creep tests.

It should be emphasized that the relaxation test is not a substitute for the creep test. However, it does provide a check on the accuracy of the law assumed for the constant-stress creep data and provides additional information which assists the metallurgists in establishing more exact analytical models to describe the deformation process.

Many of the structural problems that arise in high-temperature design stem from the occurrence of time-dependent plastic deformation. A material's resistance to deformation can be measured by a variety of test types such as the constant-stress creep test and the stress relaxation test. The present discussion deals primarily with the relaxation characteristics of a common nickel-base alloy, Inconel (nominal composition of 15 per cent chromium, 5 per cent iron, balance nickel). As used in this report, relaxation means the replacement of an initial elastic strain with plastic strain through creep. Since only the elastic strain can transmit stresses, the relaxation process can be measured through changes in the stress required to maintain a constant total strain.

As in the case of creep, the phenomenon of stress relaxation is time and temperature dependent and assumes importance in the performance of materials under a number of types of stress conditions. The data produced by relaxation testing can be applied directly to problems where stresses are maintained by elastic strain. Some of the typical design applications involving the use of elastic strain to maintain stress levels above a minimum working stress are the use of bolts to sustain flange seals, shrink or press fits, rolled-in tubes, and springs.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1. 1960.

[1] Metallurgy Division, Oak Ridge National Laboratory (operated for the U. S. Atomic Energy Commission by the Union Carbide Corp.), Oak Ridge, Tenn.

In these examples, lowering of the working stresses during service at elevated temperatures due to relaxation could impair the service performance. Likewise, stress gradients caused by design geometries or by temperature gradients are subject to redistribution through relaxation so that a more uniform gradient is achieved. The rate at which this will occur is determined by the relaxation characteristics of the material. Thus, elastic stress concentration can be lowered without appreciable damage to the structure if rapid relaxation occurs. The removal of residual stresses by annealing is controlled by stress relaxation, and, therefore, relaxation data can be used to develop stress relief treatments after fabrication by welding, forging, cold working, and machining.

Relaxation testing can also be used as a sensitive method of providing reliable information as to the long-time constant stress behavior of a material. Under certain conditions, these results, through direct computation, yield exact constant-stress creep strength values. It is not proposed, however, that the relaxation test supplant the constant-stress creep test, but rather, that it provides a means for rapid evaluation of new materials or old materials under more extreme conditions. Results from both types of testing enable the metallurgist to establish more exact analytical models to describe the deformation process and they provide the designer with strength values of higher quality.

PROCEDURE

Many different testing machines and methods have been used by previous experimenters (1–5)[2] to determine the relaxation characteristics of metals; however, most of the work has been at relatively low temperatures. Since extrapolation of these results to higher temperatures does not give reliable values, actual test data must be obtained at the desired temperature. For this purpose, it was necessary to devise new experimental techniques to achieve the accuracy desired in the temperature range of 1200 to 2000 F.

The primary effect of increasing the temperature in this type of test is to increase the initial rate of relaxation. The test machine must, therefore, incorporate a low-inertia loading mechanism capable of loading a specimen at a rapid rate and then immediately reversing and unloading at a rate sufficiently rapid to maintain a constant total strain. In the case of Inconel at 1650 F, half of the initial stress is relaxed in 18 sec. A pneumatic motor, similar to a valve operator, was found to have the required features and was incorporated in the relaxation machine design shown in Fig. 1. The motor has a diaphragm of approximately 330 sq in. and is capable of applying a 10,000-lb force. A schematic drawing of the motor with the solenoid control circuit is shown in Fig. 2.

The method of controlling the load required to achieve and maintain a desired strain depends upon the flow rate of air to the motor. Because of the large volume of air in the motor, the pressure can be regulated by simple solenoid valves operating off and on. An extensometer similar to the one developed at Westinghouse (1) is used to measure strain and was modified so as to control the load. This extensometer, shown in Fig. 3, has a 120 to 1 magnification ratio and measured strain over a 6-in. gage length specimen shown in Fig. 4. The micrometer heads establish the position of electrical contact; the top contact signals for load application and the lower contact signals for the release of the load. When the extensometer makes contact, the grid bias in a grid glow relay is cut out. The relay then actuates the proper solenoid valve and thereby controls the pressure in the pneumatic motor. A view of the solenoid valve control circuit is shown in Fig. 2. By proper adjustment of the supply pressure one can obtain strain rates on loading from 0.001 to 0.1 in. per in. per min.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The furnace is shunt-wound and is controlled by a proportioning type controller capable of maintaining the temperature within ± 2 F. By proper shunting of the furnace a gradient of less than 5 F can be achieved over the 6-in. gage length of the specimen.

Before the test is initiated, it is necessary to hold the specimen at temperature until the gradient is acceptable and the furnace has reached thermal equilibrium. The test is then run by setting the micrometer screws to obtain the desired strain, regulating the supply pressure to produce the proper strain rate, and by turning on the electronic relay. The test from this point is completely automatic and requires only periodic checks of the temperature control and gradient.

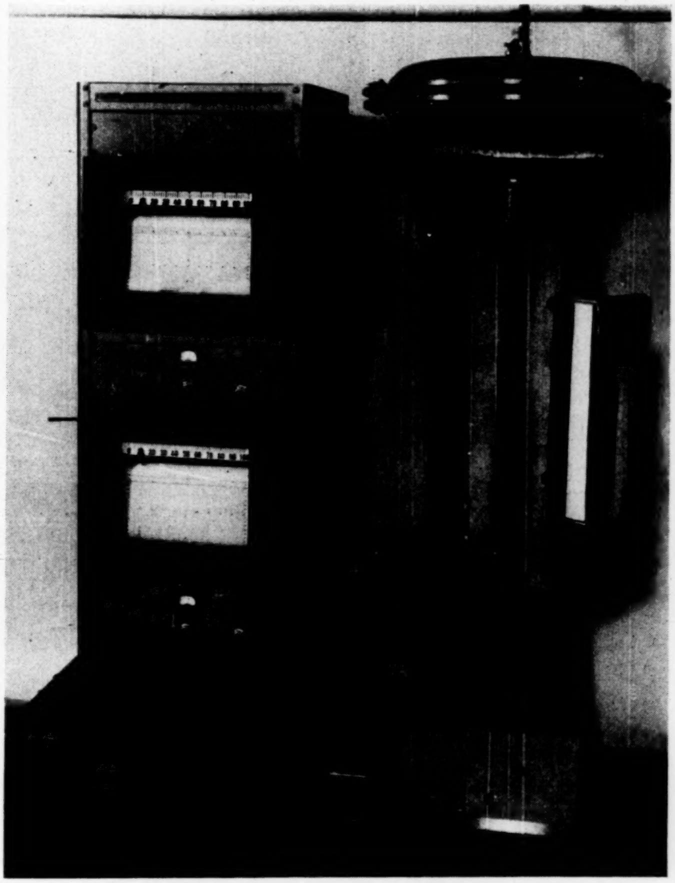


FIG. 1.—Relaxation Machine.

A load cell of the appropriate capacity is used to measure the stress applied to the specimen. The signal from the load cell is calibrated by a modified transducer calibration adapter, model Q-902, built by the Oak Ridge National Laboratory's, instrument division. The signal is continuously recorded on a strip-type potentiometer with a two-speed chart drive. A high chart-speed of 12 in. per min is used to record the first 30 min of each test and a low chart-speed of 6 in. per hr is used for the remainder of the test. In most cases, the tests are discontinued after 100 hr.

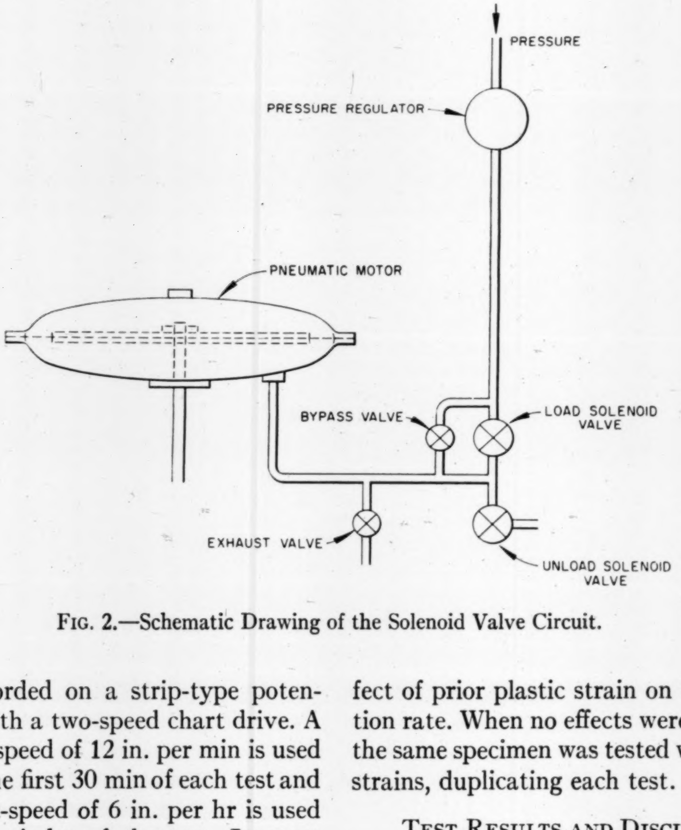


Fig. 2.—Schematic Drawing of the Solenoid Valve Circuit.

The listed results are from tests performed in Inconel, Heat NX 4364, with two grain sizes—a fine-grain size in the as-received material comparable to a 1650 F anneal, and a coarse-grained structure obtained by reannealing the as-received material at 2050 F for 2 hr. Photomicrographs of the Inconel in the two conditions are shown in Figs. 5 and 6. Most of the data were obtained from single specimens at one temperature. Each specimen was first tested at a strain of 0.05 per cent and then retested at this same strain to determine the effect of prior plastic strain on the relaxation rate. When no effects were observed, the same specimen was tested with higher strains, duplicating each test.

Test Results and Discussion

The results of relaxation testing of Inconel at temperatures of 1300, 1500, and 1650 F are shown in Figs. 7 and 8. Results shown in Fig. 7 are those obtained from testing as-received material and in Fig. 8 are those obtained from testing Inconel annealed at 2050 F for 2 hr in hydrogen. The differences between the results obtained from testing Inconel

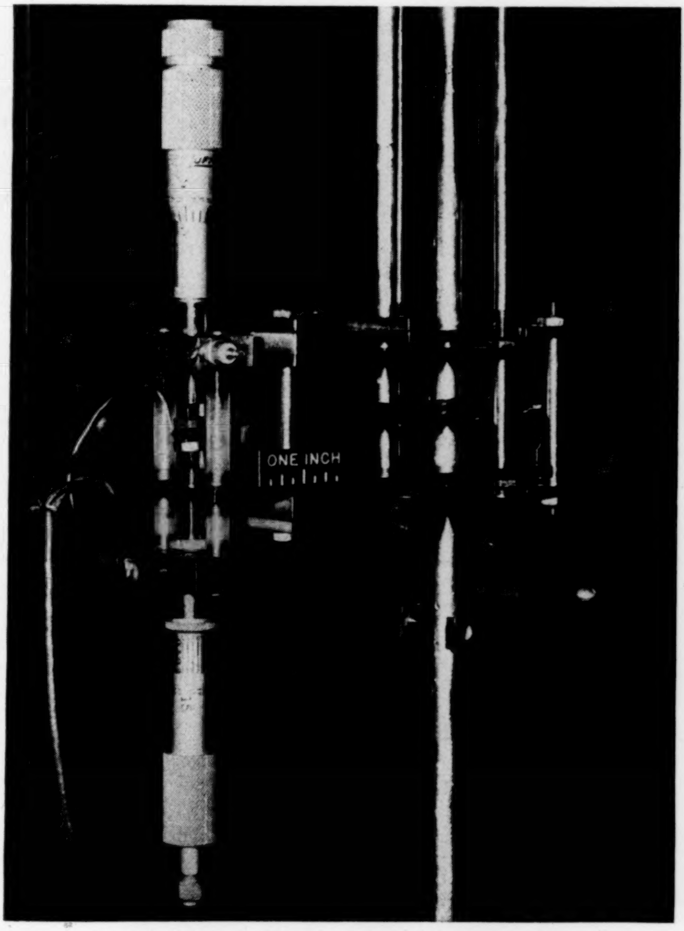


FIG. 3.—Extensometer Used in Relaxation Testing.

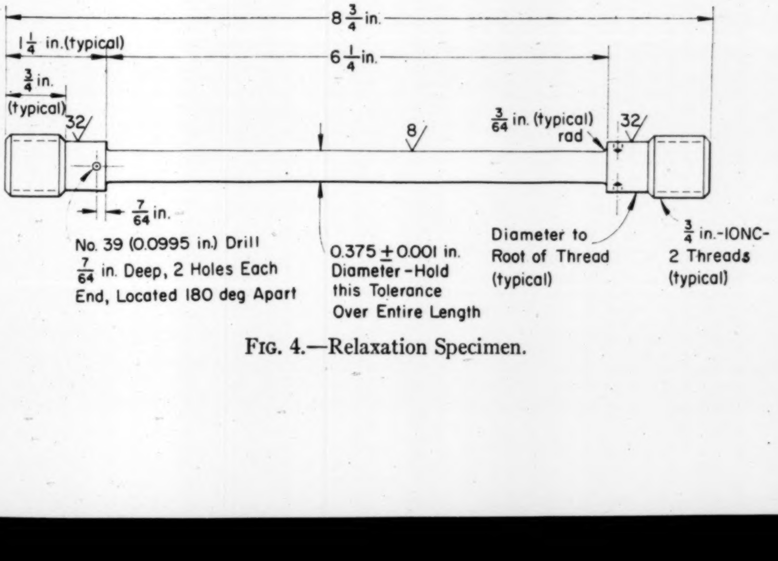

8 3/4 in.
1 1/4 in.(typical)
6 1/4 in.
3/4 in. (typical)
32
8
3/64 in. (typical) rad
32
7/64 in.
No. 39 (0.0995 in.) Drill 7/64 in. Deep, 2 Holes Each End, Located 180 deg Apart
0.375 ± 0.001 in. Diameter-Hold this Tolerance Over Entire Length
Diameter to Root of Thread (typical)
3/4 in.-10NC-2 Threads (typical)

FIG. 4.—Relaxation Specimen.

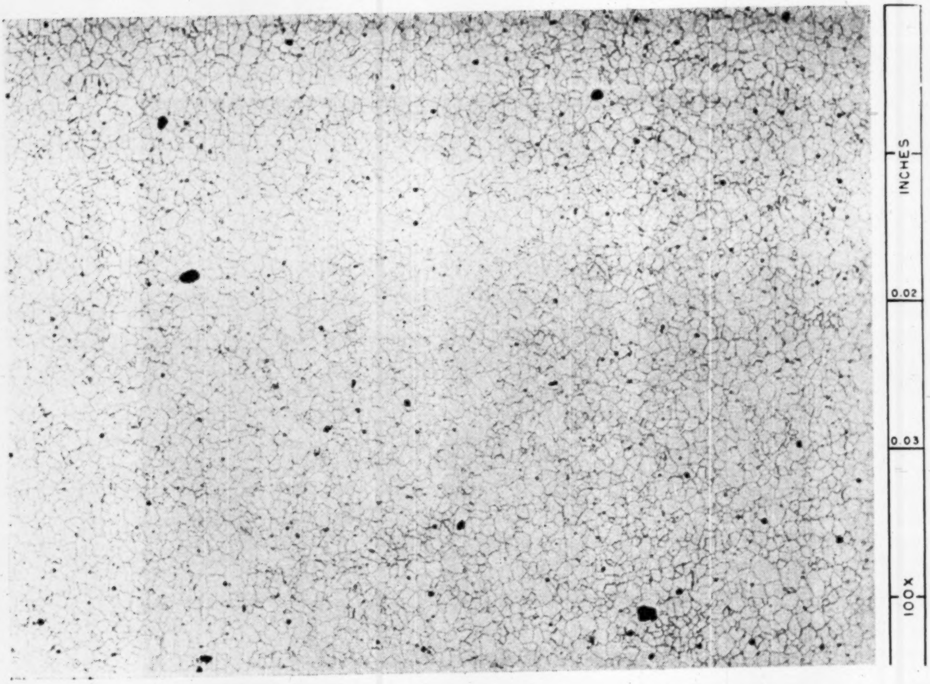

INCHES
0.02
0.03
100X

FIG. 5.—Photomicrograph of As-received Inconel.

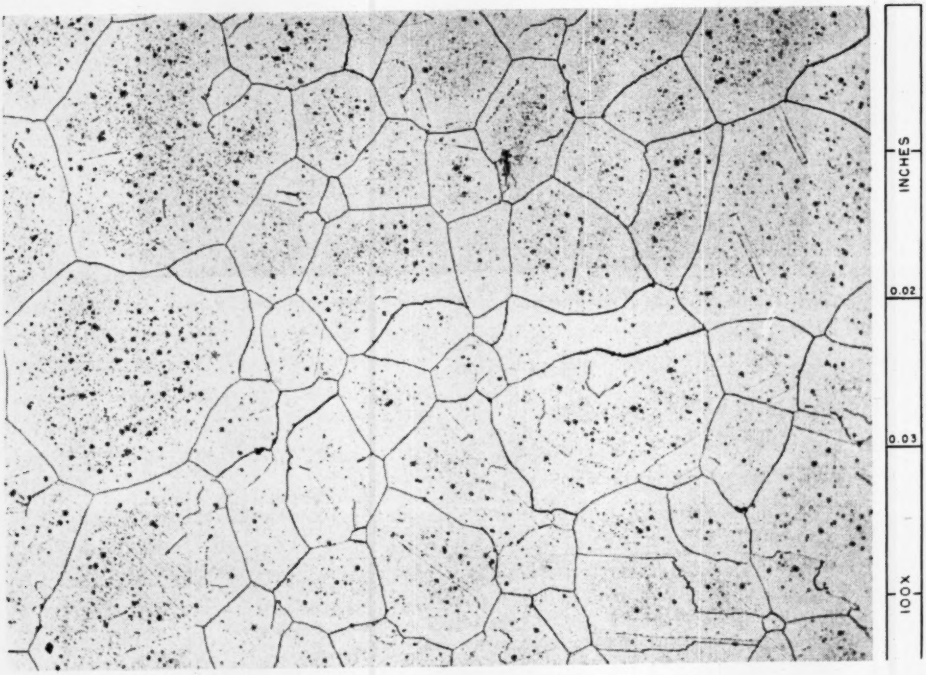

INCHES
0.02
0.03
100X

FIG. 6.—Photomicrograph of As-received Inconel Annealed at 2050 F for 2 hr.

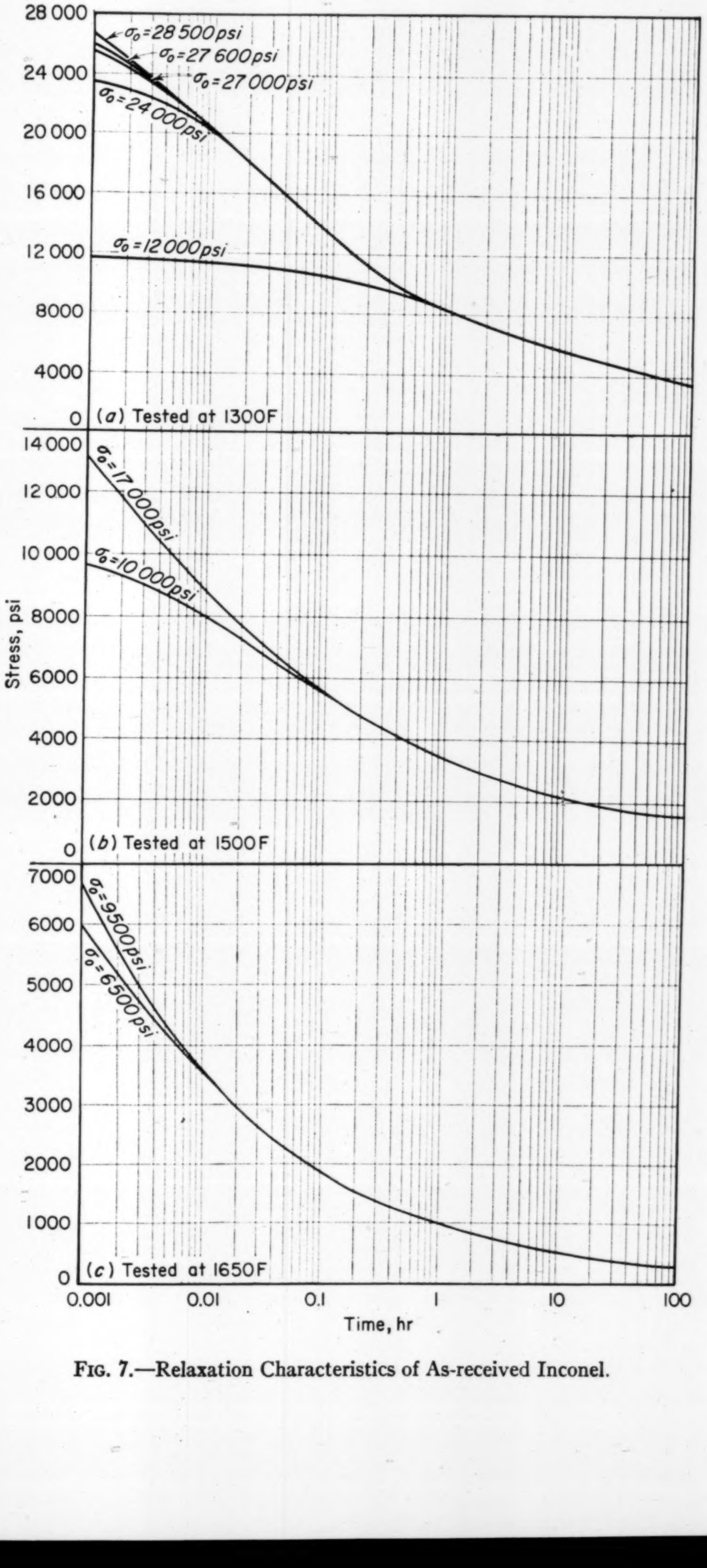


FIG. 7.—Relaxation Characteristics of As-received Inconel.

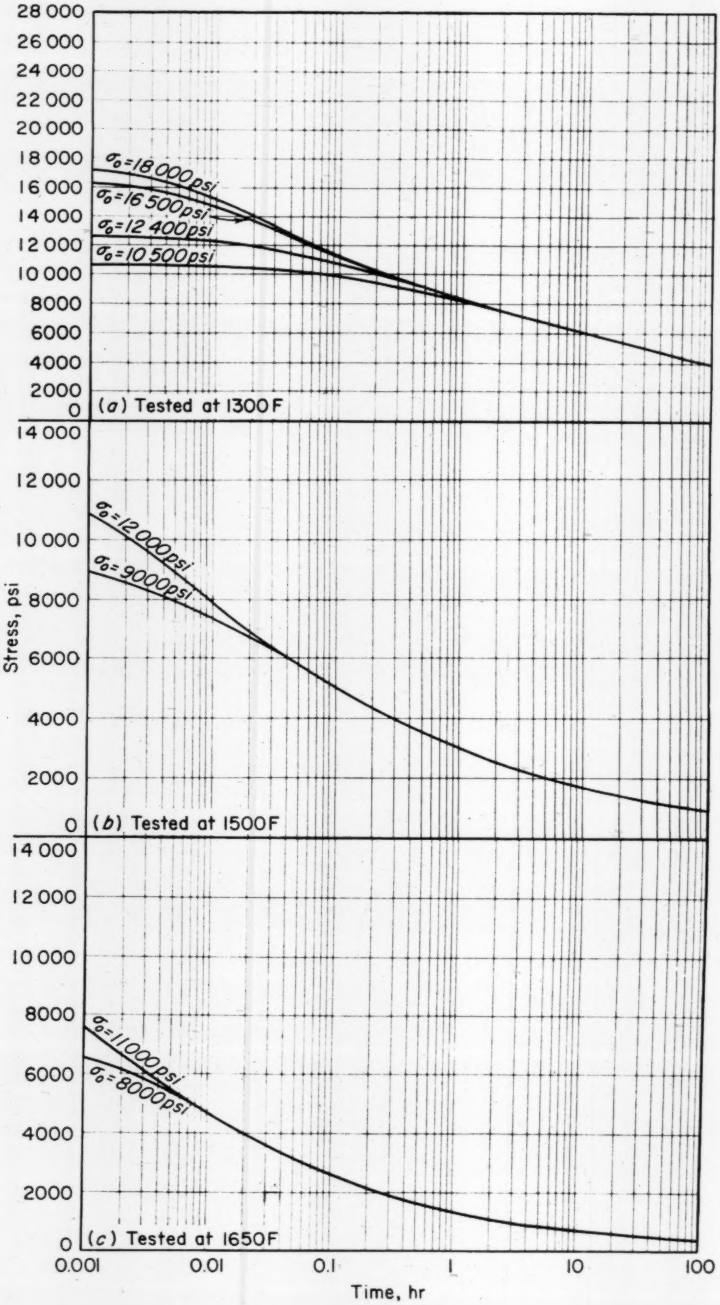


FIG. 8.—Relaxation Characteristics of Inconel Annealed at 2050 F for 2 hr

in these two conditions are not very great and, as will be shown later, less than the differences shown in creep properties.

Creep and relaxation are manifestations of the same deformation processes in metals. Although an adequate fundamental theory of creep has yet to be developed, there should exist for any given temperature an empirical law which will express the creep rate, $\frac{d\epsilon_c}{dt}$, as a function of the variables: stress, strain, and time. Both creep and relaxation measurements give only the results of the integration of such a differential law. In the past, creep test results have been used exclusively to determine these differential laws. Such tests impose the limitations of constant stress. The relaxation test has the more involved limitation of a constant total strain:

$$\epsilon_t = \epsilon_c + \epsilon_e = \text{constant}\ldots\ldots\ldots(1)$$

where:

ϵ_t = total strain,
ϵ_e = elastic strain, and
ϵ_c = plastic strain of creep.

Therefore, the determination of the differential law for creep rate by relaxation testing is extremely dlfficult. However, in many cases, a particular differential law is either known to fit the material or for ease of calculation a simple expression is used. Therefore, there are two reasons for attempting to correlate the processes of creep and relaxation: (1) to check the accuracy of the differential laws assumed for creep, and (2) to demonstrate the use of the relaxation test on a short-time determination of creep strength.

The general methods for correlating creep and relaxation data have been summarized by Roberts (6), Soderberg (7), Popov (8), and Fennie (9), and in all cases excellent agreement are found if the proper differential law is used. The one major distinction in the various expressions used for creep rate are those which include the effect of strain hardening or primary stage creep and those which ignore the primary stage of creep. One such frequently used equation which ignores the primary state of creep is

$$\epsilon_c = \left(\frac{\sigma}{A}\right)^n t\ldots\ldots\ldots\ldots(2)$$

where:

σ = applied stress,
t = time, and
A, n = material constants.

This equation, of course, does not accurately describe the creep strength of materials except under conditions where primary creep is negligible or nonexistent. For many metals as the temperature increases the degree of primary creep diminishes; therefore, Eq 2 is assumed to be accurate for high temperature. Temperatures of 1300 F and above for Inconel are sufficiently high that primary creep can be neglected; therefore, in this paper Eq 2 can be assumed to be correct.

The general method of correlating the two processes of creep and relaxation using Eq 2, originally proposed by Kanter (2) demonstrates the simplicity of using this type of equation. Since, in Eq 1 the total strain is constant, then by differentiated with respect to time, $\frac{d\epsilon_t}{dt} = 0$, and

$$\frac{d\epsilon_c}{dt} = -\frac{d\epsilon_e}{dt}\ldots\ldots\ldots\ldots(3)$$

This states that the plastic strain rate is equal to the elastic strain rate during the relaxation test. The elastic strain at any time is directly proportional to the stress divided by the modulus of elasticity, E:

$$\epsilon_e = \frac{\sigma}{E}\ldots\ldots\ldots\ldots(4)$$

Differentiating with respect to time:

$$\frac{d\epsilon_e}{dt} = \frac{1}{E}\frac{d\sigma}{dt}\ldots\ldots\ldots\ldots(5)$$

Differentiating Eq 2 with respect to time:

$$\frac{d\epsilon_c}{dt} = \left(\frac{\sigma}{A}\right)^n \quad \ldots\ldots\ldots\ldots (6)$$

Combining Eqs 3, 5, and 6:

$$\left(\frac{\sigma}{A}\right)^n = \frac{1}{E}\frac{d\sigma}{dt} \quad \ldots\ldots\ldots\ldots (7)$$

or:

$$dt = -\left(\frac{A}{\sigma}\right)^n \frac{1}{E}\, d\sigma \quad \ldots\ldots\ldots\ldots (8)$$

Equation 7 is now integrated between limits of σ_0 and σ, where the σ_0 is the initial stress at $t = 0$, and σ is the stress at time, t:

$$\int_0^t dt = -\frac{A^n}{E}\int_{\sigma_0}^{\sigma} \frac{d\sigma}{\sigma^n} \quad \ldots\ldots\ldots (9)$$

or:

$$t = \frac{A^n}{E(n-1)}\left(\sigma^{(1-n)} - \sigma_0^{\;(1-n)}\right) \ldots (10)$$

Equation 9 yields the time for an initial stress, σ_0, to relax to any given stress if the creep constants A and n are known. It is equally as easy to obtain the creep constants A and n, if the relaxation data are given. This is done by noting that when σ is small compared to σ_0, the value of $\sigma_0^{(1-n)}$ becomes insignificant providing $n > 2$ as is usually the case. Therefore, Eq 8 may be written

$$t = K\,\sigma^{(1-n)} \quad \ldots\ldots\ldots\ldots (11)$$

where:

$$K = \frac{A^n}{E(n-1)} \quad \ldots\ldots\ldots\ldots (12)$$

Using Eqs 11 and 12, the values of A and n can readily be obtained from the relaxation results. The data plotted on logarithmic coordinates should yield a straight line with a slope of $(1 - n)$ and a zero intercept of K.

Shown in Figs. 9 and 10 are logarithmic plots demonstrating the validity of Eq 14. The deviation of the straight line for very short values of time are areas where $\sigma_0^{(1-n)}$ is significant and alters the straight line. The values of A and n obtained from the relaxation results are given in Table I. The strain rate *versus* stress curves thus established by the relaxation data are compared in Figs. 11 and 12 to constant stress creep

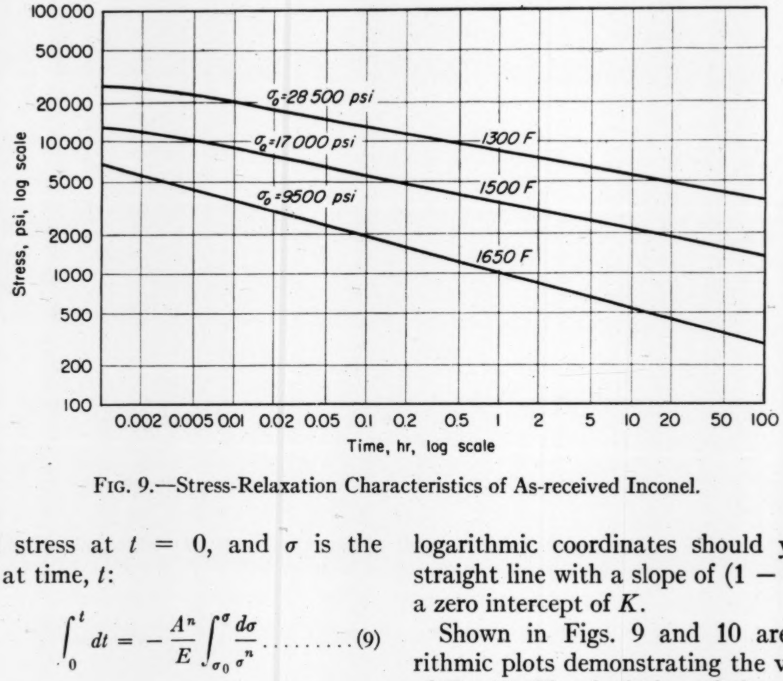


FIG. 9.—Stress-Relaxation Characteristics of As-received Inconel.

data obtained by testing fine- and coarse-grained Inconel 0.060 in. sheet, heat NX 8004. These data, obtained from testing NX 8004 in argon, have been reported previously, including the testing procedure (10). The agreement obtained is outstanding, realizing that the comparison is made for two heats of Inconel with different specimen geometries. The one exception is the comparison of properties of the fine-grained Inconel at 1650 F.

The lack of agreement between the data at 1650 F demonstrates that Eq 2 is not the correct differential law for this material and temperature. Examination of the creep curves of fine-grained NX 8004 at 1650 F, shown in Fig. 13, demonstrate that the creep rates are not linear, but are accelerating with strain or time. When the slope of the creep curve is greater than one, the creep rate is accelerating, and when it is lower than one, the creep rate is decelerating. Generally, this type of behavior indicates a condition of metallurgical instability. One fairly common type of structure instability develops when a metal is tested at temperatures higher than the temperature at which it was annealed. In this particular case the fine-grained Inconel was given what is called an equivalent 1650 F anneal. This treat-

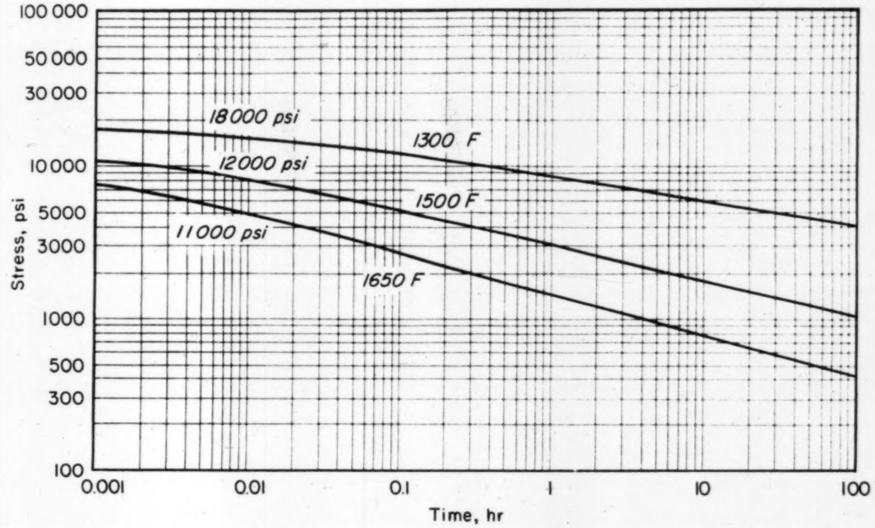


FIG. 10.—Stress-Relaxation Characteristics of Inconel Annealed at 2050 F for 2 hr.

TABLE I.—VALUES OF CREEP CONSTANTS DETERMINED BY CONSTANT-STRESS CREEP TESTING AND BY RELAXATION TESTING.

Test Type	Heat-Treatment	Test Temperature, deg Fahr	Creep Constants	
			A	*n*
Creep	[a]	1300	43 000	6.18
Relaxation			38 700	6.31
Creep	[a]	1500	20 500	5.95
Relaxation			20 100	5.81
Creep	[a]	1650	16 800	4.70
Relaxation			10 800	4.61
Creep	[b]	1300	31 600	6.85
Relaxation			30 700	7.37
Creep	[b]	1500	17 900	5.50
Relaxation			19 600	5.42
Creep	[b]	1650	10 500	5.20
Relaxation			13 100	4.79

[a] As received.

[b] Annealed at 2050 F for 2 hr.

ment is given to the material by the vendor and consists of a short-time exposure at 1850 F. Apparently this treatment does not produce a structure which remains stable under stress at 1650 F. This instability can be corrected by a higher temperature anneal as shown by the results of testing Inconel annealed at

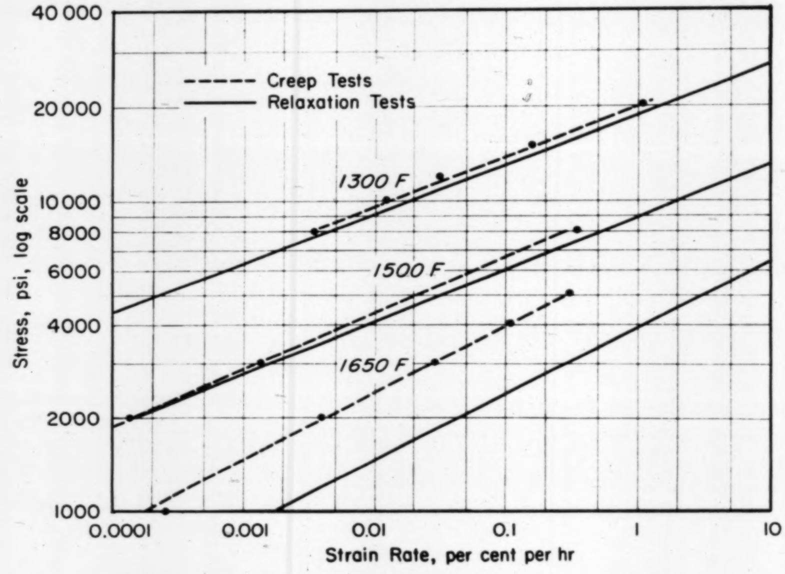


FIG. 11.—Stress *versus* Strain-Rate for As-received Inconel.

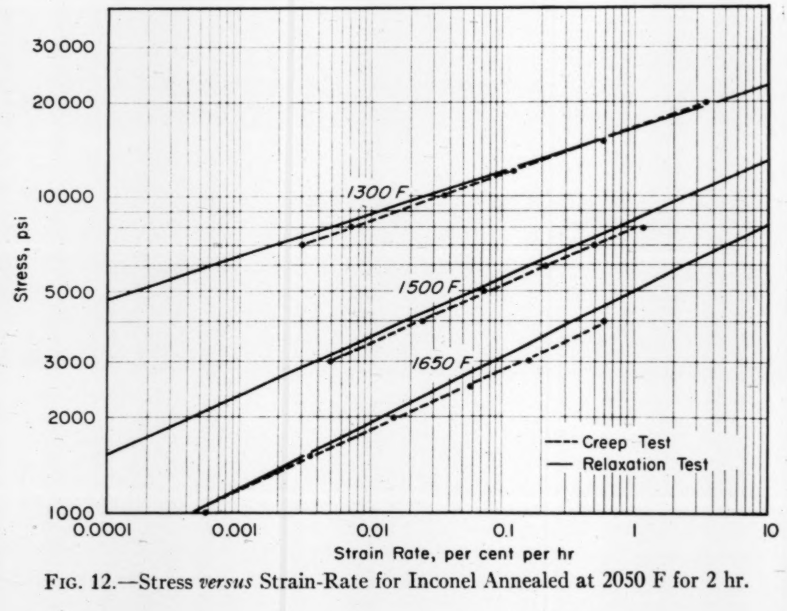


FIG. 12.—Stress *versus* Strain-Rate for Inconel Annealed at 2050 F for 2 hr.

2050 F for 2 hr. When this is done, the 1650 F results demonstrate a very close agreement using Eq 2 as shown in Fig. 12.

Although this equation or others which ignore the primary stage of creep are accurate only for specific conditions, the simplicity of the expressions warrants its use in many complex design problems where the use of the exacting solutions are sometimes unmanageable. Also, in view of the many indeterminate factors influencing creep, these solutions seem adequate. It should be remembered that the values of the creep constants, A and n, determined by constant stress creep tests, always tend to produce optimistic values of the creep strength. This has been demonstrated by the test results above for materials tested with an unstable structure. Less conservative values will be obtained by using the second-stage creep rate values where materials exhibit large strain in the primary stages. It would appear that the values of the creep constants obtained by relaxation testing, which include the effect of primary creep, may be more indicative of the creep strength of the material.

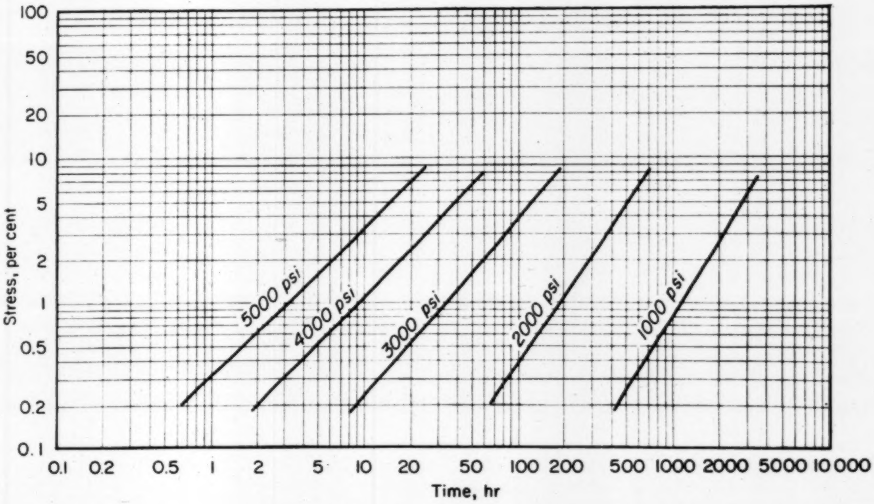


FIG. 13.—Creep Curves of Inconel Sheet (NX 8004, as-received) Tested at 1650 F in Argon.

SUMMARY

A new stress relaxation machine capable of testing materials at elevated temperatures has been developed. This machine has the advantages of low cost, dynamic response, and a control system capable of detecting a strain of 5×10^{-6}. Results produced by testing Inconel in the temperature range from 1300 to 1650 F are given.

The stress relaxation properties have long been used to provide information for the design of bolted flange joints or shrinkage assemblies operating at elevated temperatures. In addition, the data can be used to indicate the effectiveness of stress relief treatments. In this paper it is proposed that by direct computation of the relaxation test results, one can obtain reliable creep rate values.

The relaxation test therefore has the unique advantages of (1) obtaining reliable creep data over a wide range of stresses in a relatively short time and with a single specimen, (2) furnishes a check as to the accuracy of the law assumed to fit constant stress creep data, and (3) furnishes a complete stress-strain history which is recorded automatically. It is not proposed that the relaxation test will surplant the constant stress creep test, but rather than it provides a means for rapid evaluation of new materials or old materials under more extreme conditions.

Acknowledgment:

The authors wish to acknowledge the contribution of B. McNabb, Jr., in the construction of the relaxation machine and in the performing of the tests. The metallography was done by N. M. Atchley of the Metallography Section.

REFERENCES

(1) A. Nádai and S. Boyd, "Relaxation of Steels at Elevated Temperatures. A New Automatic Relaxation Machine," *Journal Applied Mechanics, Transactions,* Am. Soc. Mechanical Engrs., Vol. 60, Sept., 1938, p. A-118.

(2) J. J. Kanter, "Interpretation and Use of Creep Results," *Transactions,* Am. Soc. for Metals, Vol. 24, Dec., 1936, p. 900.

(3) E. L. Robinson, "Metals at High Temperature—Test Procedure and Analysis of Test Data," *Journal of Applied Mechanics, Transactions,* Am. Soc. Mechanical Engrs., Vol. 55, pp. 145–150 (1933).

(4) John H. M. Draper, "Creep Relaxation Testing—Tests at Constant Strain and Decreasing Load," *Engineering Journal,* May 6, 1955, pp. 564–565.

(5) W. Barr and W. E. Bordgett, "An Accelerated Test for the Determination of the Limiting Creep Stress of Metals," *Transactions,* Inst. Mechanical Engrs., Vol. 122, pp. 285–297 (1932).

(6) I. Roberts, "Prediction of Relaxation of Metals from Creep Data," *Proceedings,* Am. Soc. Testing Mats., Vol. 51, pp. 811–825 (1951).

(7) C. R. Soderberg, "The Interpretation of Creep Tests for Machine Design," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 58, pp. 733–743 (1936).

(8) E. P. Popov, "Correlation of Tension Creep Tests with Relaxation Tests," *Journal of Applied Mechanics, Transactions,* Am. Soc. Mechanical Engrs., Vol. 69, pp. A-135 (1947).

(9) I. Fennie and W. H. Heller, "Creep of Engineering Materials," McGraw-Hill Book Co., Inc., New York, N. Y. (1959).

(10) J. R. Weir, D. A. Douglas, and W. D. Manly, "Inconel as a Structural Material for High-Temperature Fused-Salt Reactor," ORNL-2264, Oak Ridge Nat. Laboratory, June 4, 1957.

DISCUSSION

Messrs. W. R. Johnson[1] and R. D. Crooks[1] (*presented in written form*).—We are pleased to see the results of this careful work on stress relaxation testing and the development of new equipment, for in the design and manufacture of springs we have been concerned with stress relaxation for many years. The tester design appears to offer real promise as a means of obtaining dynamic results, particularly in the initial rapid relaxation phase. We have attempted to analyze the general trend of the authors' data by comparing them to our experimental results on springs. Because the spring is often one of the last elements to be designed, space limitations can be so restrictive that high design stresses are unavoidable. We always need more design information at higher service temperatures, but unfortunately most of the published data are on tension creep at higher temperature, lower stresses, and much greater strain than can be tolerated in springs. Furthermore, spring designers are largely concerned with torsion or bending, where a nonuniform distribution exists across the section. This seriously complicates the experimental problems, analyses, and correlation of test data.

Experimental data on load loss in springs under constant strain can be plotted in such a way that the stress relaxation curves for different stresses fall as a family of parallel straight lines. The accompanying Fig. 18 is taken from our current paper, "The Performance of Springs at Temperatures above 900 F."[2] Relaxation appears to consist of two portions—a very rapid initial phase representing plastic yielding and a slower phase representing plastic and anelastic flow. The rate of flow seems to be constant regardless of applied stress; the initial phase is dependent upon the initial stress. Many tests have confirmed that the family of curves of varying initial stress will be parallel but displaced according to their initial stress. The authors' data from Figs. 7 and 8 when replotted in a similar way (Fig. 19) show a similar trend. It is heartening to see the agreement in plot shape, although the results on different materials and different test methods cannot be directly compared.

We have extrapolated our relaxation data on compression springs when forced to do so with several mental assurances: Relaxation is self-limiting in the sense that the stress is steadily decreasing. Also, a compression spring cannot be strained indefinitely as can a tension specimen, for when the spring goes solid, that is the end of the strain increase. We would question, however, the prediction of creep data from stress relaxation data, for relaxation may not predict the onset of the third stage of creep. More specifically, we question the reliability of short-time evaluation tests for spring applications, even in stress relaxation. Our experience with hot-set springs, in which a favorable residual stress system has been deliberately induced, is that there are two distinct stages of relaxation. In the first stage a very low rate of relaxation or even negative relaxation (recovery) may occur, but after some time the residual stresses are relieved and the spring relaxes at the same rate as if it had not been hot set. So far we are unable to predict when this second and more rapid stage will begin, except by actual experiment. This situation is considerably more complex if loading and unloading cycles at temperature must be considered.

---

[1] Assistant Director of Research and Research Metallurgist, respectively, Associated Spring Corp., Research Center, Bristol, Conn.

[2] W. R. Johnson and R. D. Crooks, "The Performance of Springs at Temperatures above 900 F," *Paper T 44*, Soc. Automotive Engrs. (1960).

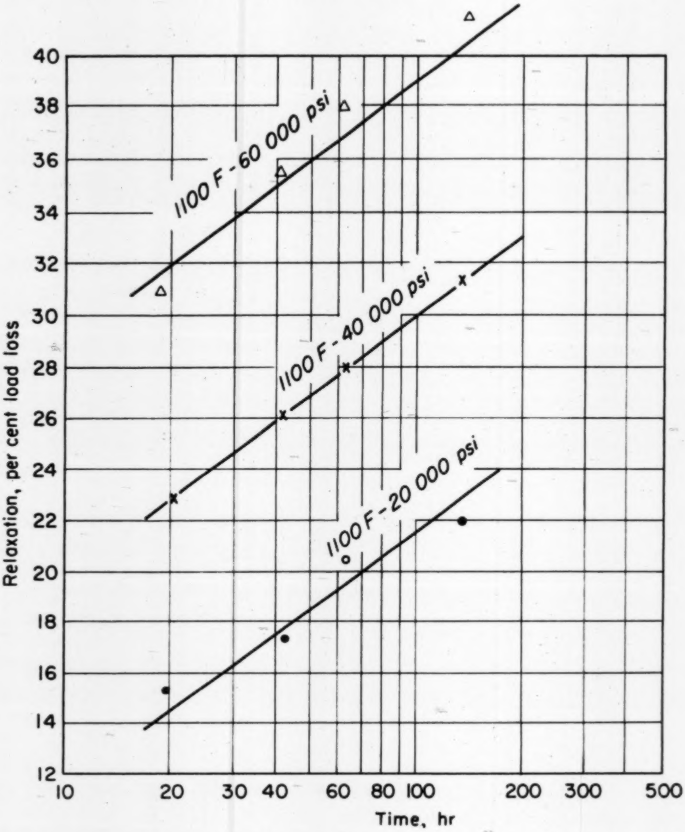


FIG. 18.—Stress Relaxation of Springs of Inconel X Alloy at 1100 F.

MR. C. R. KENNEDY (*author's closure*).—Before answering the specific questions put forth as to the usefulness of the relaxation test, I should like to discuss Messrs. Johnson and Crooks' analysis of their data and ours. The method of plotting data used by the discussers may be a convenient form for their particular application; however, the accuracy with which they plot the data and their conclusions should be examined closely. The straight lines shown by the discussers do in fact approximate certain portions of the 1300 F relaxation curve as shown in the accompanying Fig. 20(*a*). The discussers probably are not interested in load losses above 60 per cent; thus, their curves did not show a second break. It is also very interesting to note that the discussers obtained straight lines for the 1650 F data by plotting only two points. A curve taken from the data is compared with their two-point straight lines in Fig. 20(*b*). It should be pointed out that any curve, regardless of the irregularity, can be approximated by straight lines if an adequate number of lines are used. This obviously demonstrates the danger of generalizing with insufficient care or data. The author would also like to disagree entirely with the statement that: "The rate of flow seems to be constant regardless of the applied stress . . ." The per cent loss scale has a different value of stress for each initial stress curve; therefore, parallel lines on this plot definitely do not indicate a constant flow rate. Nor do the curves indicate a constant rate of load loss with time since the per cent loss is plotted versus log time.

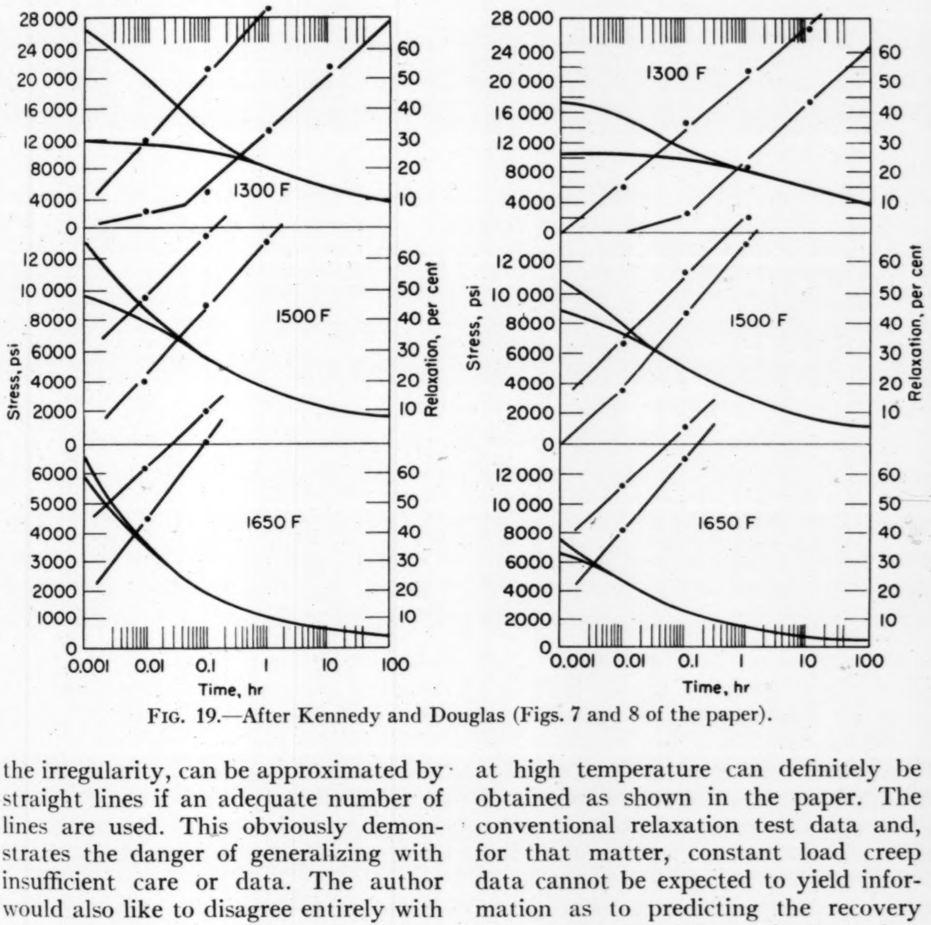
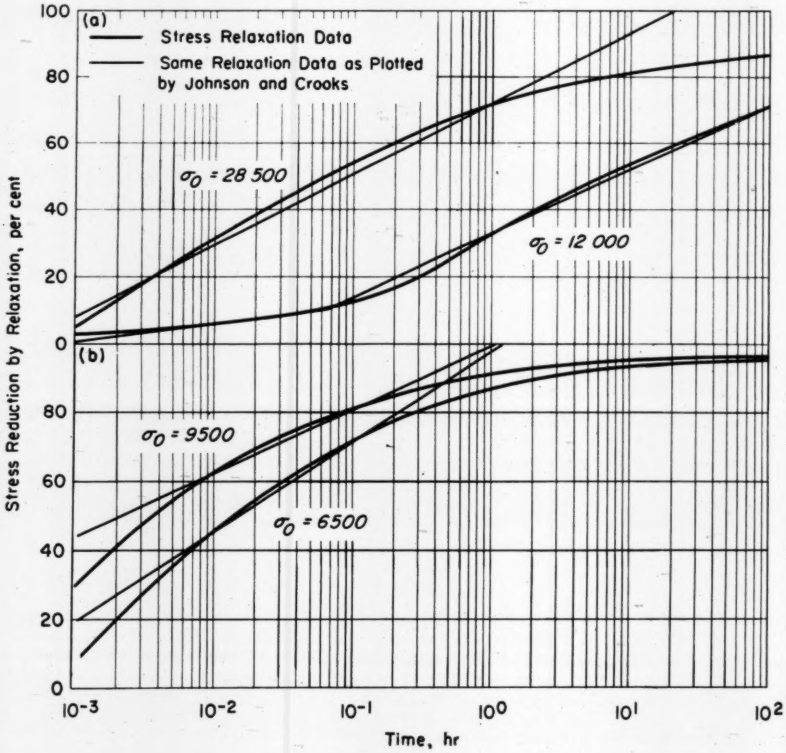


FIG. 19.—After Kennedy and Douglas (Figs. 7 and 8 of the paper).

Now for the specific questions put forth by Messrs. Johnson and Crooks. The author agrees that the entire creep curve to rupture cannot be resolved from relaxation data; however, creep rate data at high temperature can definitely be obtained as shown in the paper. The conventional relaxation test data and, for that matter, constant load creep data cannot be expected to yield information as to predicting the recovery rates obtained by "hot setting" a spring. However, the uniaxial relaxation test can be programmed to simulate this condition by prestraining the specimen similar to the spring and then obtain relaxation data applicable to their particular problems. The applicable uniaxial relaxation data can then be applied to complex stress conditions through the relationships demonstrated by Kennedy, Douglas, and Harms.[3]

The relaxation data given for Inconel in the paper are directly applicable in predicting the behavior of springs under constant restraint or under constant load conditions. The creep constants obtained from the relaxation data can be utilized in the manner outlined by Marin and Cuff[4] or Schulte[5] to determine the stress distribution under either bending or torsional moments. Thus, the rate of the moment decay can be determined from the relaxation data.

(*a*) At 1300 F.



(*b*) At 1650 F.

FIG. 20.—Stress Relaxation of As-Received Inconel.

The author agrees with the statement that results of testing different materials by different methods cannot be correlated except through empirical metallurgical relationships. However, it is our firm belief that results from different test methods on the same material can and should be directly compared. Only in this way will the frontiers in engineering be advanced and the Edisonian approach discarded.

---

[3] C. R. Kennedy, W. O. Harms, and D. A. Douglas, "Multiaxial Creep Studies on Inconel at 1500° F," *Journal of Basic Engineering*, Vol. 81, Series D, No. 4, Dec., 1959.

[4] Joseph Marin and George Cuff, "Creep-Time Relationships for Polystyrene Under Tension, Bending, and Torsion," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 1158 (1949).

[5] Charles A. Schulte, "Predicting Creep Deflections of Plastic Beams," *Proceedings*, Am. Soc. Testing Mats., Vol. 60, p. 895 (1960).

CREEP-RUPTURE PROPERTIES OF A CARBON STEEL AND A LOW-ALLOY STEEL AT 1200 TO 1800 F*

By P. N. Randall[1]

Synopsis

Creep-rupture data for carbon and low-alloy steels are needed for predicting the degree of distortion and the possibility of rupture of equipment occasionally overheated to temperatures of 1200 to 1800 F. Published data for these steels generally do not go beyond 1200 F, and extrapolation to higher temperatures is uncertain because of the phase change that occurs between 1300 and 1600 F. Therefore, data have been obtained for a carbon steel (ASTM Specification A 201, grade A)[2] and a low-alloy steel (ASTM Specification A 387, grade C)[3] at ten temperatures between 1200 and 1800 F under a stress of 2500 psi. The tests were run on constant-stress equipment, in both air and vacuum.

For both steels, the time to rupture decreased steadily with increasing test temperatures until a minimum was reached at 1400 to 1450 F; thereafter, it rose sharply to 1600 F, then again decreased. The effect on creep rate was similar to that on rupture life. Both steels were much less ductile at temperatures above 1600 F than below it. The superiority of the low-alloy steel for service at temperatures up to 1200 F disappeared at the higher test temperatures.

Contrary to prevalent belief, no evidence was found that the phase change itself weakened these steels. Apparently, the austenitic phase, present above 1600 F, is simply stronger than the ferritic phase, which predominates below 1450 F. Tests in which the temperature of the stressed specimen was cycled through the critical range verified this concept.

Short periods of extreme overheating sometimes occur in elevated-temperature service. For example, pressure vessels that have internal insulation to keep the shell temperature down may be exposed to the full temperature of the contents for a few hours if the insulation fails. Regenerator cyclones for catalytic cracking units in oil refineries are another example. Recent design specifications require that these cyclones withstand 1200 F continuously, 1500 F for two weeks each year, and 1800 F for two days each year, without collapsing. No published data are known from which to predict the serviceability of carbon or low-alloy steels for these conditions.

If high-temperature strength is required, carbon steel normally is not used at metal temperatures above 1000 F

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Research and Development Department, Standard Oil Co. (Ind.), Whiting, Ind.

[2] Tentative Specification for Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201–57 T), 1958 Book of ASTM Standards, Part 1, p. 506.

[3] Tentative Specification for Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 387–57 T), 1958 Book of ASTM Standards, Part 1, p. 635.

(1).[4] For temperatures up to 1200 F or where higher allowable stresses are needed, a low-alloy steel, such as 1.25 per cent chromium, 0.50 molybdenum, 0.75 silicon, may be used (1). These temperature limits have been established for prolonged service, largely on the basis of experience. Published data on the creep properties of carbon steels do not go beyond 1200 F, but some data on rupture properties are given up to 1400 F (2). For most low-alloy steels, the published data do not go beyond 1200 F, but for the 1.25 per cent chromium, 0.5 molybdenum steel there are some data up to 1400 F (3). Extrapolation to higher temperatures is highly uncertain, because of the phase change from ferrite-plus-pearlite to austenite that occurs, under equilibrium conditions, between about 1300 and 1600 F. The phase change itself often is believed to have a weakening effect, because it requires some atom mobility at the interface between the two phases, which might promote plastic flow or rupture under stress (4).

TABLE I.—PROPERTIES OF STEELS TESTED.

	Carbon Steel	Low-Alloy Steel
Carbon, per cent	0.17	0.13
Manganese, per cent	0.65	0.55
Phosphorus, per cent	0.009	0.016
Sulfur, per cent	0.022	0.026
Silicon, per cent	0.26	0.54
Chromium, per cent	0.08	1.21
Molybdenum, per cent	0.05	0.55
TENSILE PROPERTIES[a]		
Tensile strength, psi	64 000	80 000
Yield strength, psi	43 000	56 000
Elongation in 2 in., per cent	41	33
Reduction of area, per cent	66	72

[a] At room temperature.

Creep-rupture data have been obtained for a carbon steel and a low-alloy steel at ten temperatures in the range 1200 to 1800 F, with emphasis on the phase-transformation region. In addition, a brief investigation was made of the changes in creep rate in a stressed specimen of carbon steel as it was heated through the transformation range. A stress of 2500 psi was used in all tests, this being the maximum allowable stress for the carbon steel at 1000 F (1). The effect of scaling on creep-rupture properties was studied by testing both in air and in vacuum (0.015 mm Hg).

EXPERIMENTAL

The materials tested were: a carbon steel (ASTM Specification A201, grade A) firebox quality,[2] and a low-alloy steel (ASTM Specification A387, grade C) firebox quality,[3] nominally containing 1.25% chromium, 0.50 molybdenum, and 0.75 silicon. Chemical compositions and tensile properties at room temperature are given in Table I. The carbon steel was received as a 6 by 14-ft piece of 1-in. plate, and specimen blanks 1 in. wide by 9 in. long (in the direction of rolling) were sawed out and normalized at 1625 F before machining. The low-alloy steel was received as a 3 by 10-ft piece of $\frac{7}{8}$-in. plate, and similar specimen blanks were normalized at 1650 F and tempered at 1200 F for 1 hr before machining. For both steels, test specimens were 0.505 in. in diameter, buttonhead type; details of preparing them have been reported (5).

The constant-stress creep-rupture equipment and the test procedure used have been described (5). For the tests in air at 1300 to 1800 F, the practice of holding the specimen at temperature overnight, prior to loading, was modified to minimize scaling. The specimen temperature was held overnight at 1200 F, where the necessary adjustments were made to obtain a uniform temperature along the length of the specimen. They then were heated to the test temperature and loaded after about 1.5 hr. The button heads of the specimens were chromium-plated to prevent fouling of the pull rods with scale from the specimens.

---

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

The specimens tested in vacuum were enclosed in a stainless-steel bellows, the ends of which were welded to the shoulders of the specimens. An absolute pressure of about 0.015 mm Hg was maintained by continous pumping. The surfaces of most of the specimens remained bright, but leaks developed in the bellows in two tests at 1500 and 1600 F; hence, no vacuum tests were run at 1700 or 1800 F. Creep data were not obtained during the vacuum tests because the bellows interfered with the attachment of extensometer rods to the specimen.

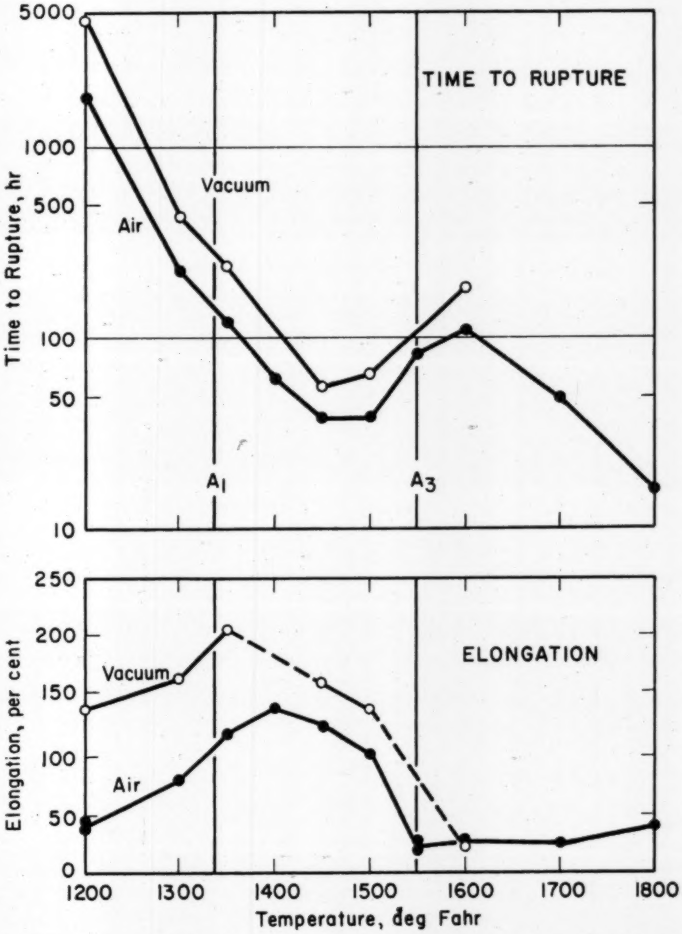


FIG. 1.—Rupture Properties of Carbon Steel Tested in Air and Vacuum Under 2500 psi Stress.

In the study of the changes in creep rate in a stressed specimen as it was heated through the phase-transformation range, the measured deformation was caused not only by creep but also by the volume change accompanying the transformation and by thermal expansion. Hence, it was necessary to repeat the test under zero stress to measure the deformation from these extraneous causes; the creep rate at several temperatures was obtained by subtraction.

RESULTS AND DISCUSSION

For the carbon steel, the effects of temperature on time to rupture and elongation are shown in Fig. 1. Time to rupture tends to decrease with increasing temperature, but the trend has two significant features. First, it continues steadily downward as the temperature passes the lower limit of the transformation range (A_1), seemingly unaffected by it. Second, it reaches a minimum between 1450 and 1500 F, followed by a sharp rise to 1600 F, where the transformation to austenite is complete (A_3), and then resumes the downward trend. Elongation increases sharply with increasing temperature from 1200 to 1400 F, as expected, but the significant feature is the

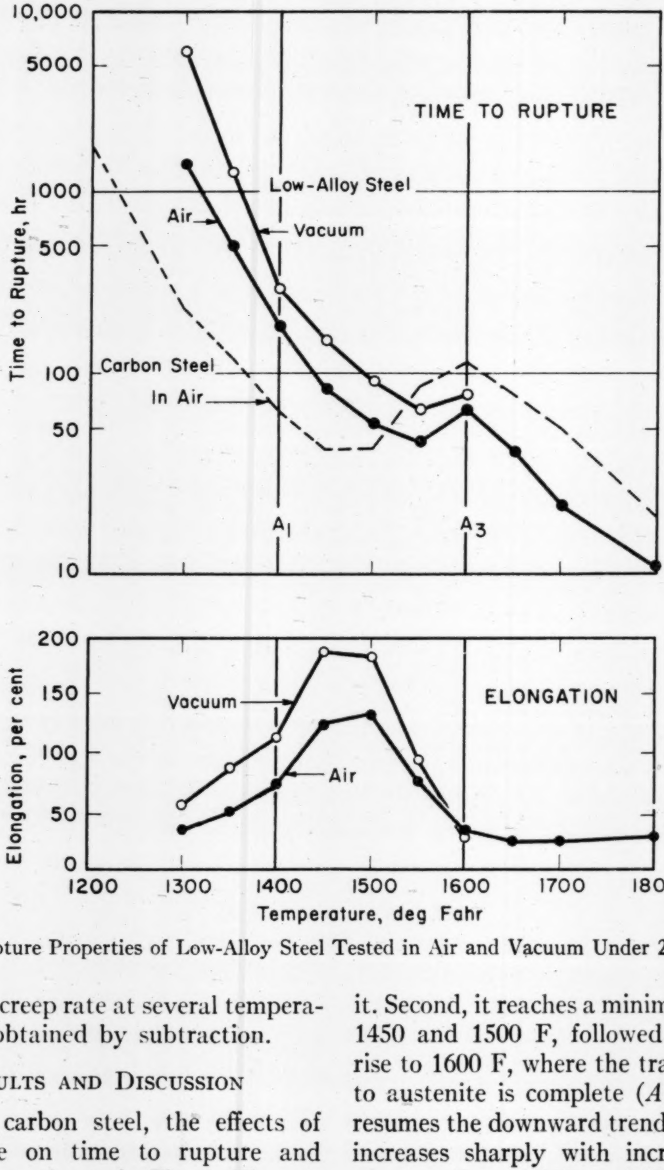


FIG. 2.—Rupture Properties of Low-Alloy Steel Tested in Air and Vacuum Under 2500 psi Stress.

sharp drop-off to a rather low constant value at 1550 F and above.

The effect of oxidation in these tests is shown by comparing the data for tests in air and in vacuum. Scaling had the most effect on elongation at 1200 and 1300 F, probably because the time of exposure was greatest at these temperatures. The ratios of the rupture lives in vacuum and in air were substantially the same at all temperatures; the increased rate of oxidation at the higher temperatures apparently was balanced by the reduction in time of exposure. These data show that the principal damage from severe overheating of parts stressed at about 2500 psi will result from increased rate of deformation rather than from increased scaling.

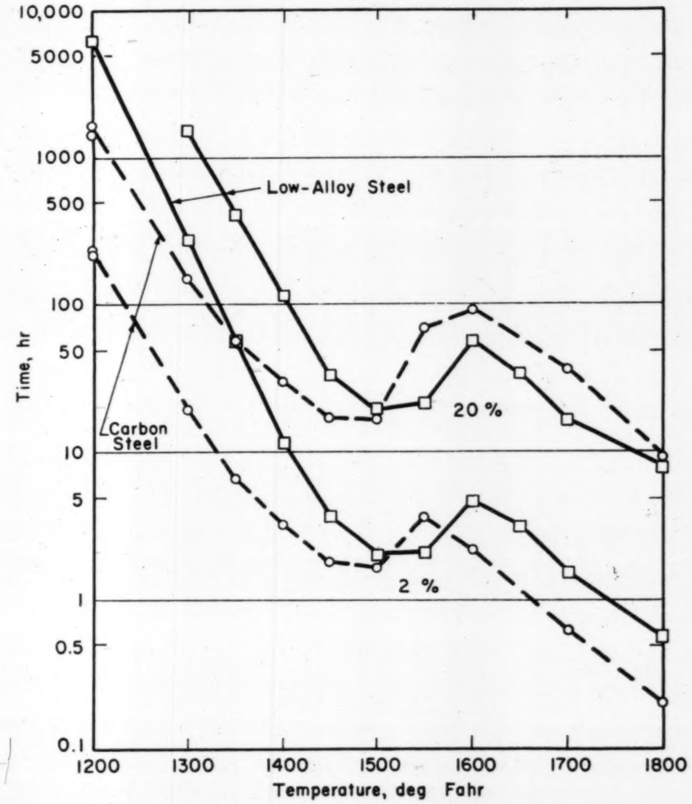


FIG. 3.—Time for Specimens of Carbon and Low-Alloy Steel to Elongate 2 per cent and 20 per cent Under 2500 psi Stress.

For the low-alloy steel, the effects of temperature on time to rupture and elongation, shown in Fig. 2, were similar to those for the carbon steel. The discontinuity in the time-to-rupture plot for the alloy steel was less pronounced than for carbon steel and occurred at a temperature about 50 F higher. Comparison of the times to rupture for the two steels shows that the known superiority of the alloy steel for service at normal elevated temperatures disappeared at 1500 F; at higher temperatures, the carbon steel was better. The comparison is developed further in Fig. 3, which shows the times required to elongate specimens of the two steels 2 per cent and 20 per cent at each test temperature. These values were chosen to present data useful in estimating the degree of distortion caused by overheating in service.

The effect of temperature on minimum creep rate, shown in Fig. 4 for both steels, shows the same pattern as for time to rupture. The transformation to austenite increased both the creep resistance of these materials and the time to rupture by an amount equivalent to a drop in temperature of about 250 F.

In all of these tests, the specimen was brought to temperature before loading; hence, the phase change had proceeded as far as it was going to go, before the test started. The question remains: "Might the act of changing phase impair the creep resistance of a part that was under stress at the time?" The results of a creep test in which the temperature of a carbon-steel specimen was varied from 1000 to 1600 F and back to 1000 F while the specimen was stressed at 2500 psi are shown in Fig. 5. Starting at the lower left, it depicts the changes in creep rate with time as the specimen was heated to the temperatures shown. For comparison

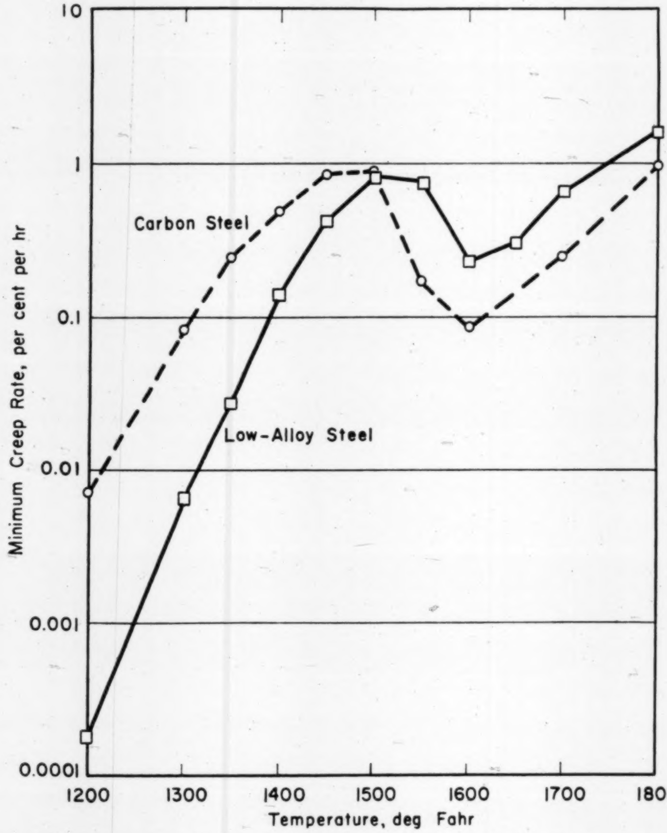


FIG. 4.—Minimum Creep Rate of Carbon and Low-Alloy Steels Tested in Air Under 2500 psi Stress.

with the maxima in creep rate that occur at about 1450 F, a plot of creep rate versus time from a previous test at this temperature also is given. Under equilibrium conditions, the phase transformation is about half completed at 1450 F; below this, ferrite predominates. Thus, the rapid acceleration in creep rate on heating from 1100 F to 1450 F must be largely an effect of temperature—particularly so because the phase change does not begin until the temperature is about 1330 F. Further heating from 1450 to 1550 F brought no increase in creep rate, apparently because the austenite, present in increasing amounts, was more creep-resistant than the ferrite it replaced. On cooling, the sudden increase in creep rate as the temperature fell from 1500 to 1450 F can be attributed to the appearance of ferrite in the structure. The maximum creep rate was remarkably close to the rate measured in the steady-temperature test a few minutes after loading the specimen. Therefore, these results give no evidence that the phase change itself promoted creep in a stressed specimen.

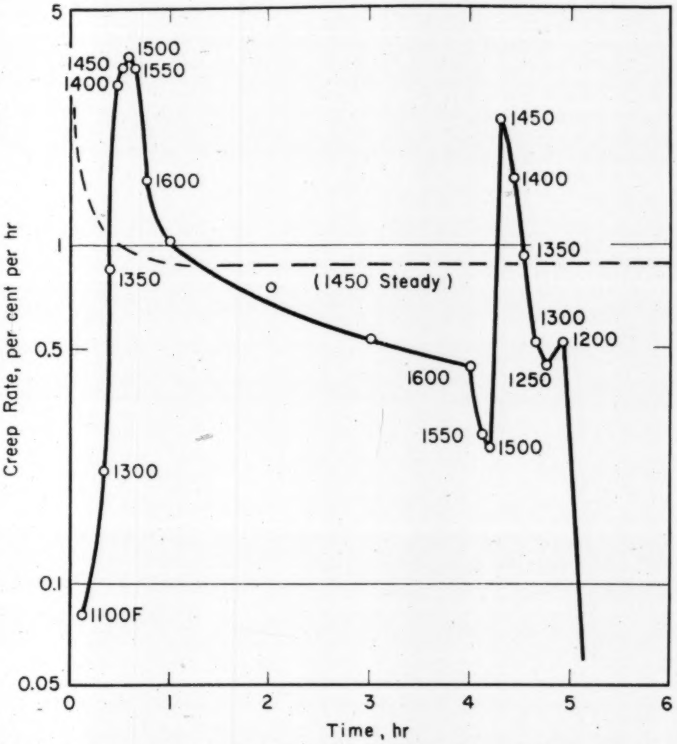


FIG. 5.—Effect of Heating and Cooling Through the Transformation Range on Creep of a Carbon-Steel Specimen Under 2500 psi Stress.

The explanation of these results also seems to fit the results of twisting tests of electrolytic iron reported by Sauveur (4). His experiment was a torsion test of a rectangular bar of electrolytic iron, which was heated at its midlength to a temperature slightly above A_3, the upper limit of the transformation range, and twisted. The most twist occurred, not where the bar was hottest, but on either side of center where the temperature was in the transformation range. They concluded that "when iron and steel undergo their A_3 transformation they become critically plastic." A better explanation seems to be that the two zones on either side of the center, in which the degree of twisting was highest, were simply the hottest zone in which the iron remained substantially all ferritic. In fact, Sauveur also presents data showing that the tensile strength was increased by the transformation to austenite.

The specimen used for this test was heated and cooled through the transformation range three times: twice under a stress of 2500 psi and once under zero stress. It then was tested to rupture at 1000 F and 10,500 psi. The time to rupture was 1640 hr—63 per cent of normal. Since the total time spent under stress in the temperature cycling tests was roughly 20 per cent of the normal life at those temperatures, the damage done to rupture life at 1000 F was not alarming.

CONCLUSION

The results of these tests are reassuring. They show that the degree of distortion and the possibility of rupture of a piece of equipment overheated in service to the austenitizing temperature is less than that predicted from the known effects of temperature in the ferritic range. No evidence was found that the phase change itself weakened the steels tested. However, the use of a low-alloy steel to guard against deformation or rupture of equipment during overheating may not be a wise practice, for its superiority over carbon steel diminished steadily with increasing temperature above 1200 F and disappeared completely at 1500 F.

REFERENCES

(1) "ASME Boiler and Pressure Vessel Code, Section VIII.—Unfired Pressure Vessels," Am. Soc. Mechanical Engrs. (1959).

2) "The Elevated Temperature Properties of Carbon Steels," Am. Soc. Testing Mats. (1955). (Issued as separate publication, *ASTM STP No. 180.*)

(3) Report on "The Elevated-Temperature Properties of Chromium-Molybdenum Steels," Am. Soc. Testing Mats. (1953). (Issued as separate publication, *ASTM STP No. 151.*)

(4) Albert Sauveur, "The Metallography and Heat Treatment of Iron and Steel," McGraw-Hill Book Co., Inc., New York, N. Y. (1935).

(5) P. N. Randall, "Constant-Stress Creep-Rupture Tests of a Killed Carbon Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 854 (1957).

DISCUSSION

Mr. J. J. Heger.[1]—Has the author any explanation as to why the carbon steel is stronger than the alloy steel at temperatures above 1500 F?

Mr. P. N. Randall (*author*).—No, I have no good explanation for it. It has been hypothesized that the carbides in the low-alloy steel had not dissolved, and, therefore it did not have the solid-solution strengthening effect of the carbon. If this were true, the superiority of the carbon steel should be greatest in the early stages of the test, and the data given in Fig. 3 show a reverse effect.

Mr. O. O. Miller.[2]—Could not the difference in carbon content between the two steels account for the difference pointed out by Mr. Heger?

Mr. Randall.—Perhaps, although it seems unlikely that the effects of the difference in carbon content (0.04 per cent) could counterbalance the effects of the molybdenum particularly, and the chromium and silicon in the low-alloy steel. Published data show that the addition of 2.5 per cent molybdenum to an austenitic stainless steel produces no strengthening at temperatures over 1650 F.

Mr. A. B. Wilder[3] (*by letter*).—The tensile, creep, and rupture properties, for a limited range of temperatures, of carbon and 1.25 per cent chromium-0.5 per cent molybdenum steels similar in chemical composition to the steels investigated by the author and shown in Table I of the paper are as follows:

Test Temperature, deg Fahr	Tensile Properties			Creep, 1 per cent in 10,000 hr, psi	Rupture, 1000 hr, psi
	Yield Strength, 0.2 per cent Offset, psi	Tensile Strength, psi	Elongation, in 2 in., per cent		
Carbon Steel					
1100....	12 500	21 000	57	1 800	6 000
1200....	9 200	15 700	63	800	2 600
1300....	6 000	10 500	69	500	1 400
1400....	4 000	7 500	80	...	...
1500....	2 000	5 500	90	...	...
1.25 Chromium, 0.5 Molybdenum Steel					
1100....	21 500	47 000	30	4 800	20 000
1200....	16 700	34 200	41	2 200	8 000
1300....	12 000	21 500	52	1 000	2 800
1400....	8 200	13 500	73	600	1 200
1500....	4 500	5 500	95	...	...

These results indicate there is no appreciable difference in strength properties of carbon and 1.25 per cent chromium-0.5 per cent molybdenum steel at the highest temperatures tested. It is quite possible 1.25 per cent chromium-0.5 per cent molybdenum steel would have slightly better corrosion or oxidation resistance. Higher chromium steels such as 5 per cent chromium-0.5 per cent molybdenum steel would have superior resistance to oxidation. The degree of oxidation is indirectly related to strength as wastage reduces the cross-sectional area of the material. Comments of the author regarding suitability of higher chromium steels for overheated regenerator cyclones would be of interest.

---

[1] Applied Research, U. S. Steel Corp., Pittsburgh, Pa.

[2] Head, Steel Section, The International Nickel Co., Inc., Bayonne, N. J.

[3] Chief Metallurgist, National Tube Division, U. S. Steel Corp., Pittsburgh, Pa.

MR. RANDALL (*author's closure*).—The comments of Messrs. J. J. Heger, O. O. Miller, and A. B. Wilder are appreciated greatly, and the data supplied by Mr. Wilder are a welcome addition to the paper.

As Mr. Wilder points out, the low-alloy steel should be superior in service where corrosion or oxidation aggravate the failure by creep rupture. These tests were too short for oxidation to be an important factor.

Higher chromium steels have not been used for regenerator cyclones because of the cost and the problems of field welding these alloys. Also, collapsed cyclones have not shown severe loss of metal by oxidation, chiefly because the periods of overheating have been short.

Since the paper was presented, a few additional creep-rupture tests have been run on the two steels at 1500 F and at stresses of 1000, 4500, 7000, and 10,500 psi. Tensile data also have been obtained at temperatures from 1200 to 1800 F. These results support the conclusion that the superiority of the low-alloy steel over the carbon steel disappeared at 1500 F and above. The reasons for this remain in doubt.

PREDICTING CREEP DEFLECTIONS OF PLASTIC BEAMS*

By Charles A. Schulte[1]

Synopsis

A simple method is proposed for predicting beam deflections under creeping conditions from tension creep data. The results of the method are in good agreement with test data on certain plastics. It is suggested that the method may apply equally well to metals.

List of Symbols

σ = stress, psi,
σ_{ME} = maximum elastic stress at time, $t = 0$,
M = bending moment, in.-lb,
ϵ = strain, in. per in.,
ϵ_E = elastic strain,
ϵ_P = plastic strain,
ϵ_o = strain in outside fiber,
$\Delta\epsilon$ = change in strain,
y = distance from neutral axis to a beam fiber,
h = half height of beam,
b = width of beam,
l = length of beam over which bending deflection is measured,
δ_m = deflection at center of span,
t = time,
K = slope of stress versus strain rate curve in steady-state creep,
o = subscript o refers to outside fiber of beam,
C = point of intersection of elastic and steady-state creeping stress distribution, and
$\dot{\epsilon}$ = creep rate.

Much attention has been devoted to the problem of predicting from ordinary tension creep data the deflections of a beam in bending. The complete solution of the problem requires a total creep law for the material in question. If we limit ourselves to constant temperature, the total creep law will be expressed in terms of four variables: creep rate, total creep strain, time, and stress. The proper formulation of such a law is generally a complicated task involving laborious effort for the determination of empirical constants. It is, in fact, a special problem in relaxation in which the creep behavior of the material in tension and compression is but one of several limiting considerations.

The hypothesis presented in this paper is an approximate method by which the deflections of creeping beams can be computed very simply. It has been checked against certain available information in the literature, both experimental and theoretical, and found to be in good agreement therewith.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

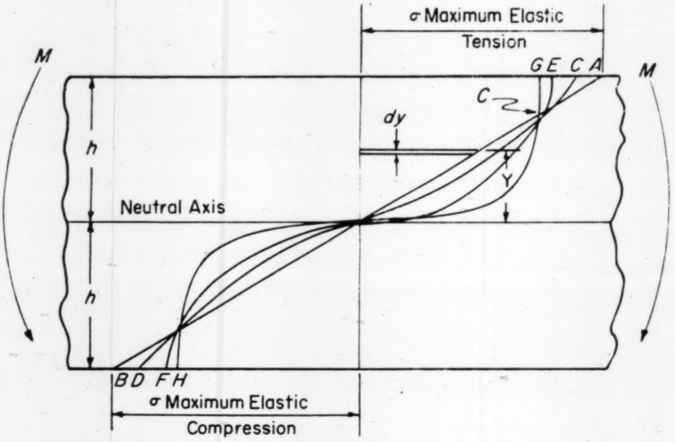
[1] Arthur D. Little, Inc., Cambridge, Mass.

CONDITIONS AND ASSUMPTIONS

In addition to satisfying the creep characteristic of the material, the relaxing beam is subject to the following additional conditions:

1. Planes before bending remain planes after bending;
2. The summation of the forces over the beam cross-section must equal zero;
3. The summation of the bending moment over the cross-section must equal the applied bending moment;
4. The limiting stress distribution under creeping conditions is the perfectly plastic stress distribution, where the maximum plastic stress equals two thirds the elastic stress.

In addition to these four conditions, it will be assumed further that we are dealing with a material whose creeping behavior is the same for both tension and compression, except for the sign of the stresses and deformations. For simplicity, we will make the additional assumption that there is, for the materials in question, a secondary creep phase wherein the change in strain is linear with time. Later, it will be shown that when this last assumption is relaxed, the accuracy of the method may not be seriously compromised. These conditions and assumptions can be applied without appreciable error to a large class of engineering materials, and in the following analysis, such materials will be considered.



Distribution *AB* at Time $t = 0$ Elastic
Distribution *CD* at Time $t = t_1$ Plastic Transient
Distribution *EF* at Time $t = t_2$ Plastic Transient
Distribution *GH* at Time $t = t_3 - \infty$ Plastic Steady State
Point *C* Lies at Intersection of *AB* and *GH*

FIG. 1.—Stress Distribution in a Beam Creeping in Pure Bending.

BASIC CONSIDERATIONS

Consider a beam in pure bending (Fig. 1). At time $t = 0$, the instant the bending moment is applied, an elastic stress distribution is assumed to exist. This is represented by line *AB*. Immediately thereafter, creeping of the beam fibers results in a partially plastic stress distribution. The outer fibers lose some of their stress by relaxation while stresses in the inner fibers increase. After time interval t_1, the stress distribution is defined by *CD*. At the end of the time interval t_2, stress distribution *EF* prevails; finally, *GH* is reached, which is invariant with time and which corresponds to steady-state creep.

During all phases of bending, planes remain plane. In the transient phase, this means that the total strain (elastic plus plastic strain) in any fiber of the beam is proportional to its distance from the neutral axis. That is, for both the outside and inside fibers, the algebraic sum of the change in elastic strain plus the creep strain with time is proportional to the fiber distance from the neutral axis. This can be expressed as

$$\frac{(\Delta\epsilon_E + \Delta\epsilon_P)_1}{(\Delta\epsilon_E + \Delta\epsilon_P)_2} = \frac{y_1}{y_2} \quad\ldots\ldots\ldots\ldots(1)$$

where the subscripts 1 and 2 refer to two different fibers in the beam. When steady-state bending is reached, $\frac{\Delta\epsilon_E}{\Delta t} = 0$ for all fibers and we have the relationship

$$\frac{\Delta\epsilon_{P1}}{\Delta\epsilon_{P2}} = \frac{y_1}{y_2} \quad\ldots\ldots\ldots\ldots\ldots(2)$$

Before the steady state is reached, fibers in the outer part of the cross-section, away from the neutral axis, will relax, finally coming to a lower stress than existed at time $t = 0$. On the other hand, fibers close to the neutral axis will gain in stress. The intermediate and final steady-state stress distributions of Fig. 1 show this. The crux of the present method lies in the significance of the point C in Fig. 1, where the initially elastic and final plastic-elastic stress distributions intersect. The assumption is now made that the stress at point C does not change from time $t = 0$ to any subsequent time. In other words, the stress distributions intermediate between the original elastic and final steady-state conditions all pass through point C. It is not argued that this is rigorously true. But the condition that the moment of all forces over the cross-section must equal the external moment applied to the beam and the fact that abrupt changes and irregularities in the stress pattern with time must be avoided make it unlikely that the stress at point C will greatly change during the primary creep phase.

If the fiber at point C remains at constant stress throughout the entire period of bending, it is always in the same condition as a tension specimen at the same stress in an ordinary creep test. Therefore, the total elongation of the fiber at C on the tension side of the beam, or contraction on the compression side of the beam at any time t can be determined from the total elongation curve of an ordinary tensile specimen at the same temperature and stress as this fiber.

If we know the strain of this fiber, ϵ_C, and its position relative to the outside surface of the beam, we can compute the strain ϵ_o in the extreme fibers using the condition that planes remain plane by the relation:

$$\epsilon_o = \frac{h}{y_C}\,\epsilon_C \quad\ldots\ldots\ldots\ldots\ldots(3)$$

With this value of ϵ_o, the deflection in the middle of the beam is

$$\delta_m = \frac{l^2\epsilon_o}{8h} \quad\ldots\ldots\ldots\ldots\ldots(4)$$

Generally, for a family of creep curves at a given temperature, a plot of creep rate (in steady-state creep) versus stress can be represented on a log-log or semilog plot as a straight line. Knowing this relationship and conditions 1, 2, and 3, we are able to establish the stress distribution, GH, and the point of intersection, C, of the elastic and steady-state distributions of bending stress. Applying Eqs 3 and 4, we can then solve the problem.

BEAM NOT IN PURE BENDING

For a beam in bending other than pure bending, if it is sufficiently long so that

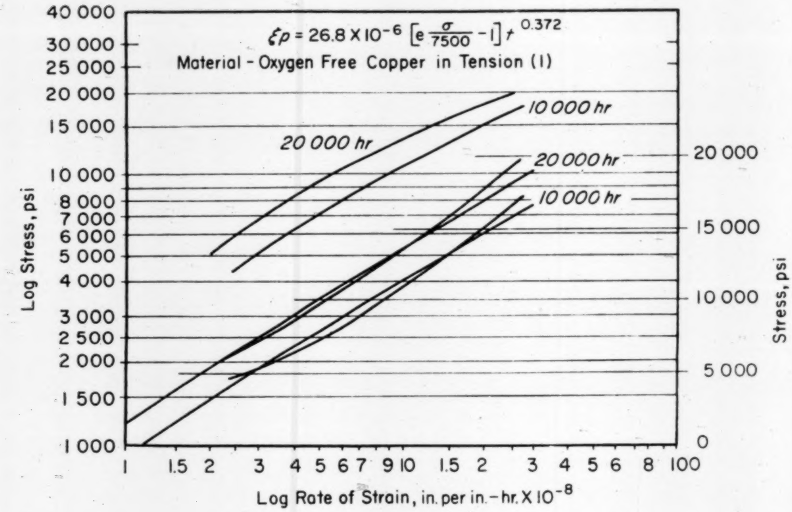


FIG. 2.—Strain Rate versus Stress (1).

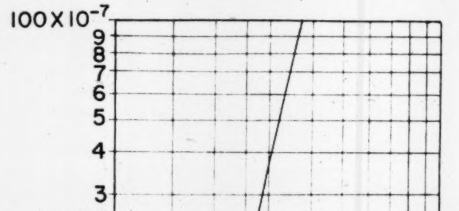


FIG. 3.—Log-Log Creep Rate-Stress for Steady State Tension (4).

shearing stresses are relatively low, the actual structure could probably be approximated by assuming it is the sum of a series of small finite lengths, each of which is in pure bending. Each such small length can then be treated in the manner described.

MATERIALS WHICH DO NOT HAVE A CONSTANT SECONDARY CREEP RATE

Some materials—for example copper—do not pass from a transient creep phase, where the creep rate continuously decreases with time, to a steady-state phase characterized by a minimum creep rate. These would have continuously varying stresses, and point C as previously determined would not exist. However, if we take a sufficiently long time, we can approximate fairly well a log-log or semilog relationship between creep rate and stress. For purposes of illustration, Fig. 2 shows log-log and semilog plots of creep rate versus stress in copper (1)[2] for 10,000 and 20,000 hr. Within rather broad stress

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

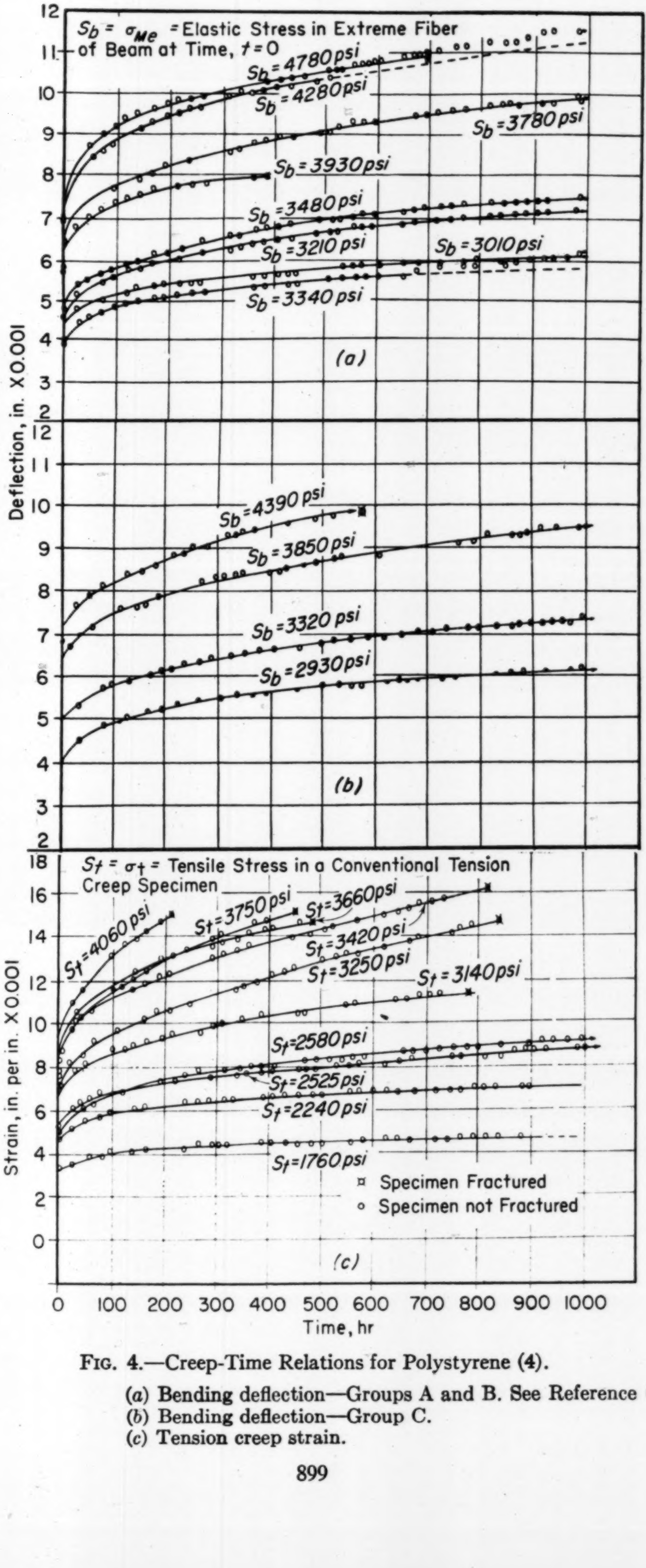


FIG. 4.—Creep-Time Relations for Polystyrene (**4**).

(*a*) Bending deflection—Groups A and B. See Reference (**4**).
(*b*) Bending deflection—Group C.
(*c*) Tension creep strain.

limits, a straight-line relation between stress and strain rate on a semilog plot for time $t = 20{,}000$ hr is a fairly accurate approximation. As will be shown, this particular approximation yields a result which agrees well with results of a beam calculation made by Popov (2,3). It appears possible, if we treat the creep data of materials such as copper in this manner, to evaluate bending deflections of beams with good approximation.

EXPERIMENTAL CHECKS

For experimental verification of the foregoing method of analysis, reference is made to a paper on "Creep-Time Relations for Polystyrene under Tension, Bending and Torsion" by Joseph Marin and George Cuff presented before the Society in 1949 (4). Figures 3 and 4 are taken from this paper.

Referring to Fig. 3, we see that a log-log relationship between creep rate and stress holds for this material under steady-state conditions.

$$\log \dot{\epsilon}_1 - \log \dot{\epsilon}_2 = K(\log \sigma_1 - \log \sigma_2) \,..\, (5)$$

$$\log 100 \times 10^{-7} - \log 10^{-7} = K(\log 3700 - \log 1350)$$

$$K = 4.6 \,\dots\dots\dots\dots\, (6)$$

For steady-state creep,

$$\frac{\dot{\epsilon}_1}{\dot{\epsilon}_2} = \frac{\sigma_1{}^K}{\sigma_2{}^K} \,\dots\dots\dots\dots\, (7)$$

Let $\dot{\epsilon}_o$ be the steady-state creep rate in an outside fiber and $\dot{\epsilon}_y$ the steady-state creep rate in any other fiber. Then, from the fact that planes remain plane, we can establish the following relation:

$$\frac{\dot{\epsilon}_y}{\dot{\epsilon}_o} = \frac{\sigma_y{}^K}{\sigma_o{}^K} = \frac{y}{h}$$

$$\sigma_y = \sigma_o \left(\frac{y}{h}\right)^{1/K} \,\dots\dots\dots\, (8)$$

where σ_o and σ_y are stresses in the outside and interior fibers, respectively, and h and y are the distances from these fibers to the neutral axis. If M is the externally applied moment, we know that

$$M = 2b \int_o^h \sigma_y y \, dy \,\dots\dots\dots\, (9)$$

Substituting for σ_y its value from Eq 8,

$$M = 2b \int_o^h \frac{\sigma_o}{h^{1/K}} y^{1/K+1} \, dy = \frac{2bh^2\sigma_o}{1/K + 2} \,\dots\, (10)$$

The maximum elastic stress at time $t = 0$ was

$$\sigma_{ME} = \frac{3M}{2bh^2}$$

or

$$M = \frac{2bh^2\sigma_{ME}}{3} \,\dots\dots\dots\dots\, (11)$$

Equating Eqs 10 and 11, and solving for σ_o, we get

$$\sigma_o = \frac{\dfrac{1}{K} + 2}{3} \sigma_{ME} \,\dots\dots\dots\, (12)$$

For the polystyrene of Marin and Cuff, $K = 4.6$, and the steady-state stress in the outside beam fibers is

$$\sigma_o = \frac{\dfrac{1}{4.6} + 2}{3} \sigma_{ME} = 0.74\sigma_{ME} \,\dots\, (13)$$

The fiber stress, σ_C, common to all states of stress can now be found in terms of σ_o from Eq 8 as follows:

$$\sigma_C = \left(\frac{y_C}{h}\right)^{1/K} \sigma_o \,\dots\dots\dots\, (14)$$

If σ_C remains constant from time $t = 0$, we may express it in terms of the initial maximum elastic stress σ_{ME} as

$$\sigma_C = \frac{y_C}{h} \sigma_{ME} \,\dots\dots\dots\dots\, (15)$$

Substituting the value of σ_o from Eq 13

TABLE I.—CORRELATION OF EXPERIMENTAL BEAM DEFLECTIONS OF FIGS. 4(*a*) AND (*b*) WITH PREDICTED DEFLECTIONS FROM FIG. 4(*c*) AND EQS 17 AND 19.

Experimental δ_m from Figs. 4(*a*) and (*b*)					Predicted δ_m (Eq 19)				
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
σ_{M_E}	100 hr	200 hr	300 hr	900 hr	σ_C	100 hr	200 hr	300 hr	900 hr
2930	5×10^{-3}	5.25×10^{-3}	5.5×10^{-3}	6×10^{-3}	1990	5×10^{-3}	5.2×10^{-3}	5.3×10^{-3}	5.9×10^{-3}
3320	5.8	6.25	6.5	7.25	2260	6	6.2	6.4	7.1
3850	7.5	8	8.3	9.4	2620	7.1	7.6	8	9.1
4390	8.25	8.85	9.3	9.8	2990	8	8.6	9.2	10
				500 hr					500 hr
3340	4.8	5.1	5.25	5.75	2270	6	6.2	6.4	7.1
3010	5.2	5.4	5.5	6	2050	5.1	5.25	5.4	6
3480	5.8	6.2	6.5	7.5	2370	6.30	6.90	7.0	7.1
3780	7.8	8.25	8.65	9.8	2560	7	7.50	7.90	9
3210	5.6	6.0	6.2	7.1	2180	5.9	6	6.2	7
3930	7.4	7.75	7.9	8.1	2680	7.3	7.8	8.1	8.25
				400 hr					400 hr

NOTE.—$\sigma_{M_E} = S_b$ Elastic stress in extreme fiber of beam in bending at time $t = 0$.
σ_C = Stress at point *C* from Eq 17.

in Eq 14, and equating Eqs 14 and 15, we solve for y_C and get,

$$y_C = 0.68h \qquad \ldots\ldots\ldots\ldots (16)$$

If we now substitute this value of y_C in Eq 15, we get

$$\sigma_C = 0.68\sigma_{ME} \qquad \ldots\ldots\ldots\ldots (17)$$

Marin and Cuff employed a beam of the following dimensions:

Cross-section width—$b = \frac{1}{2}$ in.

Depth of beam—$2h = 1\frac{1}{2}$ in.

Deflections were measured for the case of pure bending over a 2-in. length between the gage points. Therefore, $l = 2$ in.

Figures 4(*a*) and (*b*) give the bending deflection, δ_m, as a function of time for various values of the elastic bending stress in the outside fibers. Figure 4(*c*) gives tension creep strain in inches per inch as a function of time for various stress values.

To determine whether we can predict bending deflections from tension creep data, we select from Figs. 4(*a*) and (*b*) the bending deflection δ_m of the beam corresponding to an initial elastic stress σ_{ME}. This is an experimental bending deflection. To determine this deflection from tension creep data, we proceed as follows:

1. Compute σ_C.
2. Find from Fig. 4(*c*) tension creep strain ϵ_C corresponding to σ_C and time t.

From the fact that planes remain plane, we know that the total strain in the outer fibers and fibers ϵ_C can be related according to Eq 3. We also know that for pure bending δ_m at the middle of the beam $= \frac{\epsilon_o l^2}{8h}$. Therefore, substituting for ϵ_o from Eq 3 we get:

$$\delta_M = \frac{\epsilon_C \dfrac{h}{y_C} l^2}{8h} = \frac{\epsilon_C l^2}{8y_C} \qquad \ldots\ldots\ldots (18)$$

For the material under consideration,

$$\delta_M = 0.98\epsilon_C \qquad \ldots\ldots\ldots\ldots (19)$$

The accuracy of this method when applied to the data of Marin and Cuff can be seen from Table I. Column *1* gives the maximum elastic bending stress of the loaded beam in the experimental tests. Columns *2*, *3*, *4*, and *5* give the experimental creeping deflections for the beam under a bending moment to produce this initial elastic stress. Column *6* gives the critical stress σ_C at y_C. Columns *7*, *8*, *9*, and *10* give the deflections calculated from the total tension creep elongation corresponding to σ_C as shown in Fig. 4(*c*). The agreement is, in general, good.

Of further interest is the comparison of results obtained by the method outlined here and another approach to the problem of predicting beam deflections presented by Popov (2). The material used in Popov's analysis is copper, which never reaches and remains at a minimum rate of creep. It is thus one of those materials which does not conform to the assumption of a steady-state creep phase as postulated earlier for the materials to which the analysis can be made to apply. Popov (2) has given a computation of the deflection of a beam employing a stepwise method based on the strain-hardening theory of creep. He also has shown the stress distribution for various times t for which the beam deflections have been calculated. The tension creep strain versus time family of curves for this material has been defined by Popov (3) as follows:

$$\epsilon_P = 26.8 \times 10^{-6}(e^{\sigma/7500} - 1)t^{0.372}$$

By differentiating this expression with respect to time and holding σ constant, we can find the creep rate which corresponds to any particular value of t and σ. This has been done for $t = 10{,}000$ and 20,000 hr and is plotted on a log-log and semilog relation as a function of stress (see Fig. 2).

The example calculated by Popov (2) involves a maximum elastic stress of 13,500 psi in the beam. In this range of stresses, a fair approximation to a straight-line relation between stress and log creep rate can be made either for 10,000 or 20,000 hr. Therefore, if we assume steady-state creep,

$$\log \dot{\epsilon}_1 - \log \dot{\epsilon}_2 = K(\sigma_1 - \sigma_2) \quad \ldots\ (20)$$

and

$$\log \frac{\dot{\epsilon}_1}{\dot{\epsilon}_2} = K(\sigma_1 - \sigma_2) \quad \ldots\ (20a)$$

where the value of K could be obtained from either curve with reasonably good accuracy.

Using the same nomenclature as before, we find

$$\frac{\dot{\epsilon}_y}{\dot{\epsilon}_o} = \frac{y}{h} = e^{K(\sigma_y - \sigma_o)} \quad \ldots\ (20b)$$

$$\sigma_y = \frac{K\sigma_o + \log y/h}{K} \quad \ldots\ (21)$$

$$M = 2b \int_o^h \sigma_y y \, dy$$

$$= 2b \int_o^h \left[\frac{K\sigma_o + \log y/h}{K} \right] y \, dy$$

$$= bh^2 \left(\sigma_o - \frac{1}{2K} \right) \quad \ldots\ (22)$$

Equating Eqs 11 and 22 and solving for σ_o, we get

$$\sigma_o = \frac{2}{3}\sigma_{ME} + \frac{1}{2K} \quad \ldots\ (23)$$

From Eqs 15 and 20(b)

$$\frac{y_C}{h} = \frac{\sigma_C}{\sigma_{ME}} = e^{K(\sigma_C - \sigma_o)} \quad \ldots\ (15a)$$

Substituting for σ_o its value from Eq 23 into Eq 15(a)

$$K\sigma_C - \log \sigma_C = \tfrac{2}{3}K\sigma_{ME} + \tfrac{1}{2} - \log \sigma_{ME} \quad \ldots\ (24)$$

from which σ_C can be calculated when σ_{ME} and K are known.

For the present case (with reference to Fig. 2), and using Eq 20

$$\frac{21 \times 10^{-8}}{3 \times 10^{-8}} = e^{K(15{,}000 - 5{,}000)}$$

$$K = \frac{1}{5130} \quad \ldots\ (25)$$

$$\sigma_o = \tfrac{2}{3}\sigma_{ME} + 2565 \text{ (from Eq. 23)} \quad \ldots\ (26)$$

With reference to Popov's analysis, the beam assumed in his example is 6 in. deep and 10 in. long. In pure bending under a moment of 162,000 in.-lb, the extreme fiber stress is 13,500 psi. From Eq 26, $\sigma_o = 11{,}565$ psi. By substitution of values in Eq 24, we find $\sigma_C = 10{,}000$ psi. From Eq 20 (b) we find $y_C = he^{K(\sigma_C - \sigma_o)} = 0.735h$. Also from Eq 3 we know that $\epsilon_o = \dfrac{h}{y_C}\epsilon_C = 1.36\ \epsilon_C$. With these values we can compute the plastic displacement δ_m at the middle of the beam. Again, if we use Eq 4 to define the relation of δ_m and ϵ_o, we get for the beam under consideration:

$$\delta_M = \frac{\epsilon_C l^2}{5.88h} = 5.67\epsilon_C \quad \ldots\ (27)$$

Using $\sigma_C = 10{,}000$ psi and going into

TABLE II.—DEFLECTIONS OF A COPPER BEAM PREDICTED BY METHOD OF POPOV AND THE PROPOSED METHOD.

Time, hr	Plastic Strain, Tensile	Deflections	
		Popov	Eq 27
250	0.00050	0.0035	0.00284
500	0.00070	0.0043	0.00400
750	0.00085	0.0050	0.00483
1000	0.00095	0.0056	0.00540
1250	0.00111	0.0060	0.00632
1500	0.00115	0.0065	0.00654
1750	0.00121	0.0070	0.00690
2000	0.00130	0.0073	0.00740

NOTE.—The deflections given above are based on plastic strains only.

Davis' tension creep curves, one finds the values of total creep strain corresponding to the times shown in Table II. Also given in Table II are the beam deflections δ_m computed by Popov, who used his method and the values obtained by the method outlined above.

SUMMARY

In the foregoing, a hypothesis is offered along with some experimental verification. If this hypothesis is validated by additional test results, it could prove useful in simplifying the prediction of creeping deflections of beams.

REFERENCES

(1) E. A. Davis, "Creep and Relaxation of Oxygen Free Copper," *Journal of Applied Mechanics, Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, p. A-101 (1943).

(2) E. P. Popov, "Bending of Beams with Creep," *Journal of Applied Physics*, Vol. 20, No. 3, March, 1949, p. 251.

(3) E. P. Popov, "Correlation of Tension Creep Tests with Relaxation Tests," *Journal of Applied Mechanics*, Vol. 14, No. 2, June, 1947, p. A-135.

(4) Joseph Marin and George Cuff, "Creep-Time Relations for Polystyrene under Tension Bending and Torsion," *Proceedings*, Am. Soc. Testing Mats., Vol. 49 (1949).

AN EXAMINATION OF HIGH-TEMPERATURE STRESS-RUPTURE CORRELATING PARAMETERS*

By Francis J. Clauss[1]

The purpose of this paper is to examine methods for correlating high-temperature stress-rupture data presented in the literature. These correlations have been accepted by some and roundly criticized by others. Although their use can lead to large errors, the correlations are nevertheless not without value. Judiciously used, and with proper realization of the magnitude of possible errors, they furnish an excellent means of summarizing many data in convenient form for comparison and for improving the accuracy of extrapolations.

General Background

The existence of a correlation between the stress, σ, temperature, T, and time-to-failure, t_r, can be stated mathematically by the equation:

$$f(\sigma, T, t_r) = 0 \ldots\ldots\ldots\ldots (1)$$

The existence of such a function is implicit in the conventional plots of log stress against log rupture life for lines of constant temperature, or any modifications or cross-plots thereof, or any extrapolations therefrom.

Without any loss of generality, Eq 1 can be written as an explicit expression for any one of the variables in terms of the others. If the time-to-failure is selected as the dependent variable, one obtains:

$$t_r = g(\sigma, T) \ldots\ldots\ldots\ldots (2)$$

Also, without any loss of generality, any of the variables can be replaced by a function of that variable. It is convenient to use log t_r and log σ in place of t_r and σ, respectively, and write:

$$\log t_r = \psi(\log \sigma, T) \ldots\ldots\ldots (3)$$

If there were a suitable quantitative theory of solid-state behavior, one might deduce the form of the function $\psi(\log \sigma, T)$. In the absence of such theory, an empirical form of the function can be deduced from experimental observations.

Experimental Observations

In the following sections, the stress-rupture behaviors of different materials are classified according to their degree of complexity. Suitable equations are developed for the simplest type of behavior, and these are then modified systematically to handle the more complex behavior.

Class I:

The simplest type of variation of one quantity with another is that represented by a straight line. A family of straight parallel lines are often obtained when the stress and time-to-rupture at constant temperatures are plotted against each other on logarithmic scales. An example of this behavior is shown by

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26-July 1, 1960.

[1] Research Scientist, Chemistry and Plastics, Lockheed Aircraft Corp., Missiles and Space Division, Sunnyvale, Calif.

Timken 35-15 stainless steel, the data for which are plotted in Fig. 1 (**2**).[2] For this type of behavior, the function $\psi(\log \sigma, T)$ becomes:

$$\log t_r = a \log \sigma + b(T) \ldots\ldots (4)$$

where a is the slope of the isothermal lines and $b(T)$ is a measure of the "relative height" of the isothermal lines above each other. The term a is a negative-valued constant, independent of stress and temperature, in accordance with the fact that the time-to-failure decreases as the stress increases and the isothermals are straight and all have the same slope. The term $b(T)$ is a function of temperature only; its value should be positive and should decrease as the temperature increases. These terms have been determined by statistical analysis of the raw data for Timken 35-15 steel, and the following equation has been obtained:

$$\log t_r = -4.350 \log \sigma + 29.5003 - 0.007300T \ldots\ldots (5)$$

The lines in Fig. 1 were calculated according to this equation and are seen to correlate the data accurately. (The symbol T used in the figures denotes temperature. Unless otherwise noted, all values are in degrees Fahrenheit.)

One can now use Eq 5 to examine the applicability of several correlations to the same data.

The correlations of Larson and Miller (**4**) and of Orr, Sherby, and Dorn (**5**) originate in the basic Arrhenius equation (**3**). Creep is assumed to be a process that follows the same laws as chemical reactions, so that the rate of creep obeys the equation:

$$\dot{\epsilon} = A^{-E/RT} \text{ at constant stress} \ldots\ldots (6)$$

where:

$\dot{\epsilon}$ = the creep rate,
A = a constant, independent of temperature,
E = an energy change, independent of temperature,
R = the gas constant, and
T = absolute temperature.

By assuming that the time-to-rupture varies inversely as the rate of creep, one

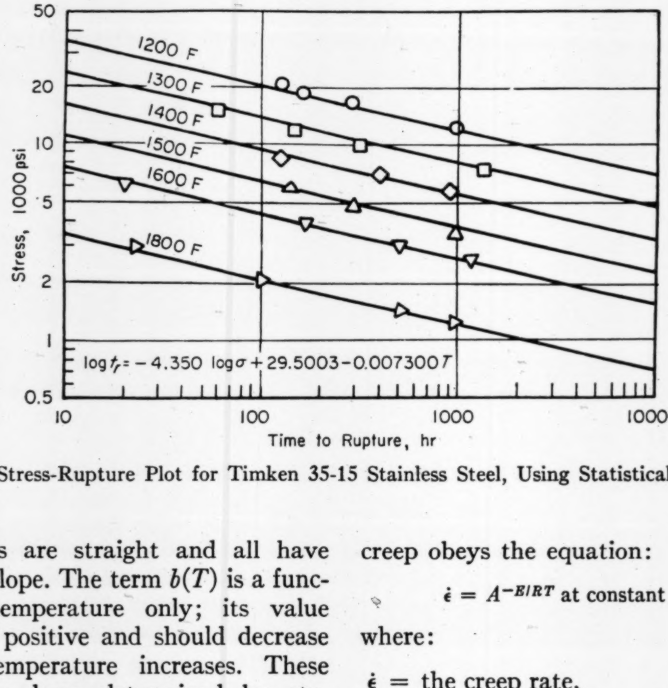


FIG. 1.—Stress-Rupture Plot for Timken 35-15 Stainless Steel, Using Statistically Determined Equation.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

obtains:

$$t_r = \frac{c}{\dot{\epsilon}} = \left(\frac{c}{A}\right) e^{E/RT}$$

$$= A' e^{E/RT} \text{ at constant stress.......(7)}$$

Temperature and time-to-rupture can be combined in a single term in two ways to furnish correlations that at first appear unrelated. Larson and Miller (4) first converted to common logarithms, so that Eq 7 became:

$$\log t_r = \log A' + \frac{E}{2.3RT} \text{ at constant stress....(8)}$$

Rearrangement gives:

$$T(-\log A' + \log t_r) = \frac{E}{2.3R} \text{ at constant stress.....(9)}$$

Orr, Sherby, and Dorn (5) used the form:

$$t_r e^{-E/RT} = A' \text{ at constant stress...(10)}$$

If the constants A' and E are independent of temperature, Eqs 7 to 10 imply that a plot of log t_r against $1/T$ should give a series of straight lines of constant stress. Moreover, when $1/T = 0$, the value of A' is t_r. Larson and Miller pointed out that their plots of log t_r against $1/T$ did approximate a series of straight lines of constant stress and that these lines extrapolated to a common point of intersection at $1/T = 0$. Because of this common point, the value of A' was independent of stress. The value of log t_r at this point was about -20 for the materials they studied, and accordingly, their parameter took the form:

$$T(20 + \log t_r) = \text{contant for constant stress...(11)}$$

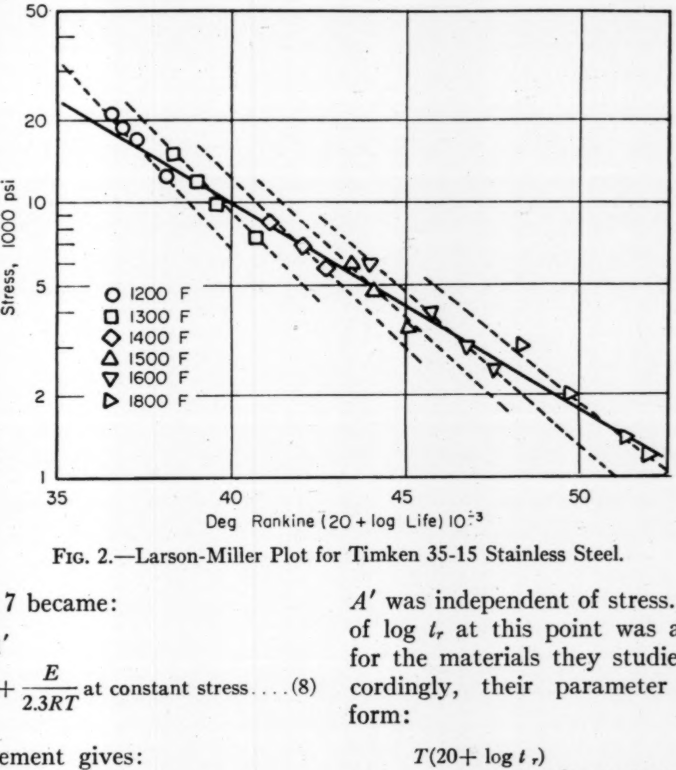


FIG. 2.—Larson-Miller Plot for Timken 35-15 Stainless Steel.

Figure 2 illustrates the use of the Larson-Miller parameter to correlate the data for Timken 35-15 stainless steel. The single solid line represents the "average" behavior about which the data points scatter, and the dashed lines connect the data points for each temperature.

The individual dashed lines in Fig. 2

were calculated from Eq 5. The fact that the data points fall closely upon these lines follows necessarily from the validity of the empirical equation, which has been demonstrated in Fig. 1. The point to be made here is that the parameter °R(20 + log t_r) does not reduce the experimental stress-rupture data to a single straight line. On the contrary, the correlation still results in a series of isothermal lines, just as in the conventional plot of log σ against log t_r (Fig. 1). Also, the time to rupture cannot be read easily and directly along the abscissa, but must be obtained from an unnecessarily complicated factor that has to be solved for each point. Finally, the correlation reduces the time scale so that it cannot be read as accurately.

The points in Fig. 2 scatter in such a way that at each temperature, the points of high stresses lie above the solid "average" line and the points of low stresses lie below this line. In other words, points of short life lie above this line and the points of long life lie below it.

It necessarily follows that any attempt to predict long-time behavior from short-time results by using the parameter °R (20 + log t_r) will introduce serious and systematic errors.

For example, the error involved in extrapolating the short-time life at 1600 F to predict the long-time life at 1400 F (an extrapolation of only 200 F at a constant stress of 6000 psi) results in a predicted life that is more than 400 per cent longer than the material would actually survive.

Despite such errors, there are occasions on which the use of the Larson-Miller parameter is justified, and it is instructive to examine the basis for such degree of correlation as may appear to exist. Essentially, the Larson-Miller parameter attempts to adjust the time-to-failure at a given stress for variations in the test temperature; in order to do this, it substitutes a correlating factor in place of time so that the set of isothermal curves on the conventional plot of log σ against log t_r is reduced to a single curve. This is indicated schematically in Fig. 3. The significance of such a correlating factor can be shown by returning to the general statement of the existence of a functional relation between

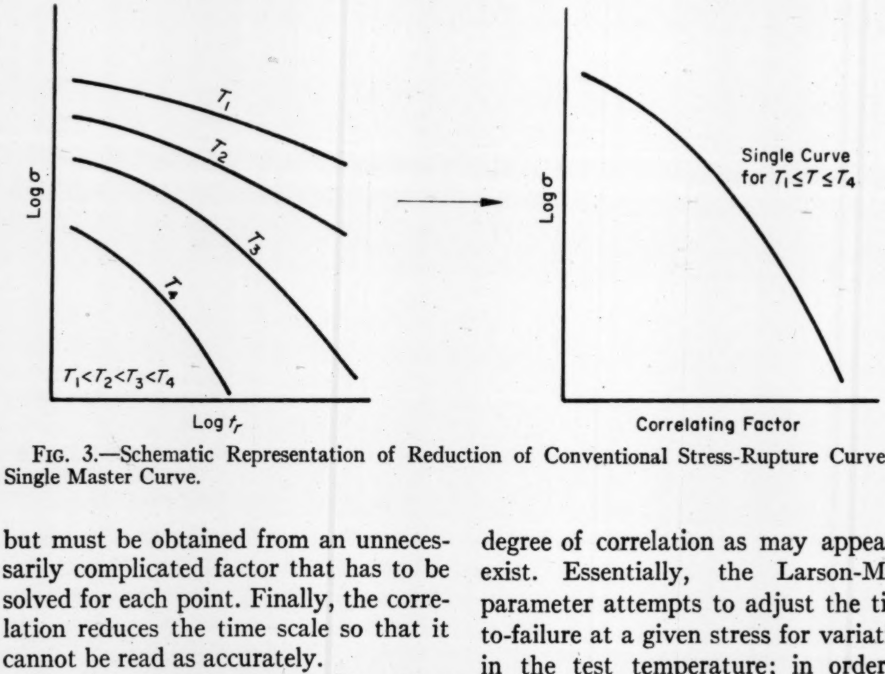


FIG. 3.—Schematic Representation of Reduction of Conventional Stress-Rupture Curves to Single Master Curve.

stress, temperature, and time-to-failure presented in Eq 3.

Differentiating Eq 3, one obtains the perfect differential:

$$d \log t_r = \left(\frac{\partial\psi}{\partial \log \sigma}\right)_T d \log \sigma + \left(\frac{\partial\psi}{\partial T}\right)_\sigma dT \quad \ldots\ldots\ldots (12)$$

where the partial differential $\left(\frac{\partial\psi}{\partial \log \sigma}\right)_T$ is the rate of change of the log of the time-to-failure with respect to the log of the stress at constant temperature, and $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$ is the rate of change of the log of the time-to-failure with respect to temperature at constant stress. The first of these is the slope of an isothermal line on the plot of log t_r against log σ, while the second measures the separation between the isothermal lines as the temperature is changed at some fixed stress. In general, both of these differentials are functions of stress and temperature.

For a correlation of the type just described, that is, a correlation of stress-rupture lives with temperature at constant stress, Eq 17 reduces to:

$$d \log t_r = \left(\frac{\partial\psi}{\partial T}\right)_\sigma dT \text{ at constant stress} \ldots (13)$$

By integrating Eq 13 between a fixed reference temperature and any temperature T, one obtains:

$$\log t_r - \int^T \left(\frac{\partial\psi}{\partial T}\right)_\sigma dT = \text{constant at constant stress} \ldots. (14)$$

This equation is the general form of the correlating parameter. Integration of the term $\left(\frac{\partial\psi}{\partial T}\right)_\sigma dT$ cannot be carried out until the differential $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$ can be evaluated. In general, this differential is a function of both stress and temperature. For the class I type of behavior, however, any pair of isothermal lines is separated by the same amount over the entire range of stresses considered (that is, the lines are parallel to each other) and $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$ is, at most, a function of temperature only.

For many high-temperature steels the separation between two isothermal lines representing a difference of 100 F is of the order of one cycle of log t_r on the conventional log σ against log t_r plot. This means that the value of the differential $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$ should be of the order of magnitude of:

$$\left(\frac{\partial\psi}{\partial T}\right)_\sigma \cong \frac{\Delta \log t_r}{\Delta T} = \frac{\log \frac{(t_r)_2}{(t_r)_1}}{T_2 - T_1} = \frac{\log 10}{-100} = -0.0100$$

and, as a first approximation, the general correlating parameter for these materials is:

$$\log t_r + 0.010T = \text{constant at constant stress} \ldots (15)$$

Returning to the Larson-Miller parameter, one can rewrite it in the form:

$$°\mathrm{R}(20 + \log t_r) = 20 \times °\mathrm{R} + \log t_r \times °\mathrm{R} \ldots (16)$$

Since the term log t_r is much smaller than 20, one can, without much change in the value of the parameter, use an average value for deg Rankine with log t_r and make the temperature correction in the first term only; that is, one can write:

$$°\mathrm{R}(20 + \log t_r) \cong 20 \times °\mathrm{R} + \log t_r \times °\mathrm{R}_{\mathrm{avg}} \ldots (17)$$

Since the temperatures of interest are from 1200 to 1800 F, roughly, or about an average value of around 1500 F, one can take deg Rankine$_{\mathrm{avg}}$ to be equal to 2000. Then:

$$°\text{R}(20 + \log t_r) \equiv 20 \times °\text{R} + 2000 \times \log t_r \quad \text{. . . (18)}$$

The difference between the values of the parameter as defined by Larson and Miller and the approximate form is only about 1 to 2 per cent. By algebraic rearrangement of Eq 18, one obtains:

$$\begin{aligned} &°\text{R}(20 + \log t_r) \\ &\quad \cong 20 \times °\text{R} + 2000 \log t_r \\ &\quad \cong 20(°\text{F} + 460) + 2000 \log t_r \\ &\quad \cong 20 \times °\text{F} + 9200 + 2000 \log t_r \\ &\quad \cong 2000(\log t_r + 0.010 \times °\text{F}) + 9200 \quad \text{. . (19)} \end{aligned}$$

The values of the coefficients 2000 and 9200 are constant and can be neglected in writing the parameter. Therefore, in place of the parameter °R(20 + log t_r), one can use the parameter (log t_r + 0.010 T), where T is the temperature in degrees Fahrenheit. Thus, by algebraic manipulation of the Larson-Miller parameter, one obtains exactly the parameter given in Eq 15, which was obtained from a general empirical approach to the problem as the first approximation for the behavior of all materials.

Figure 4 shows the degree of accuracy with which the parameter log t_r + 0.010 T approximates the data for Timken 35-15 stainless steel. This figure shows a closer degree of correlation than was obtained with the original form of the Larson-Miller parameter (Fig. 2). Figure 4 also reveals the same type of systematic errors. In the case of Timken 35-15 stainless steel, these errors are due to using an incorrect value of $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$.

A more correct value of $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$ is obtained by differentiating Eq 5 with respect to temperature at constant stress, which gives:

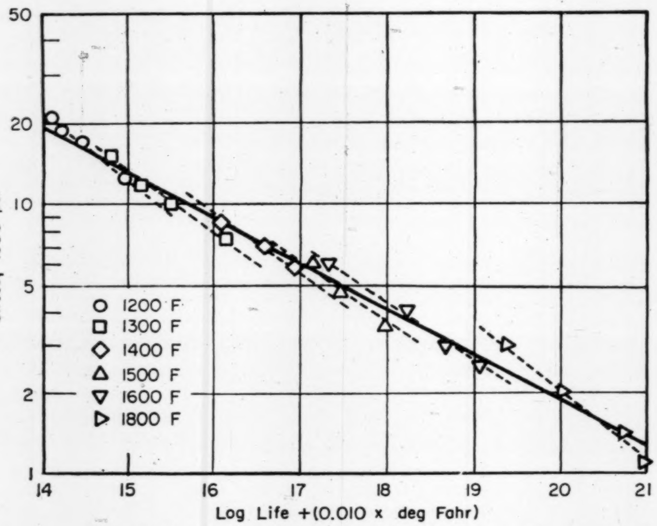


FIG. 4.—Master Curve for Timken 35-15 Stainless Steel, Using Approximate Correlating Parameter.

$$\left(\frac{\partial \log t_r}{\partial T}\right)_\sigma = \left(\frac{\partial\psi}{\partial T}\right)_\sigma = -0.007300 \quad \text{. . (20)}$$

An easier way to evaluate this differential, which avoids the mathematical development which has been detailed here, is simple to "scale off" the separation between the isothermal lines in Fig. 1. Inserting this value of the differential into Eq 14, one obtains:

$$\log t_r + 0.007300\, T = \text{constant at constant stress} \quad \text{. . (21)}$$

as the correlating parameter for Timken 35-15 stainless steel. Thus, a plot of log σ against this parameter should be a straight line, as shown in Fig. 5. Note the improvement in correlation over that shown in Figs. 2 and 4.

In the correlation of Orr, Sherby, and Dorn, the factor A' in Eq 10 is assumed to be a function of stress (rather than a constant, independent of stress, as in the correlation of Larson and Miller). Then:

$$t_r^{-E/RT} = f(\sigma) \quad \ldots\ldots\ldots\ldots (22)$$

or, in logarithmic form:

$$\log t_r - \frac{E}{2.3RT} = g(\sigma) \quad \ldots\ldots\ldots (23)$$

For Timken 35-15 steel, the log of the time-to-rupture at constant temperature varies linearly with the log of the stress, so that, for this case, Eq 23 becomes:

$$\log t_r - \frac{E}{2.3RT} = A \log \sigma + B \quad \ldots\ldots (24)$$

or

$$\log t_r = A \log \sigma + B + \frac{E}{2.3RT} \quad \ldots\ldots (25)$$

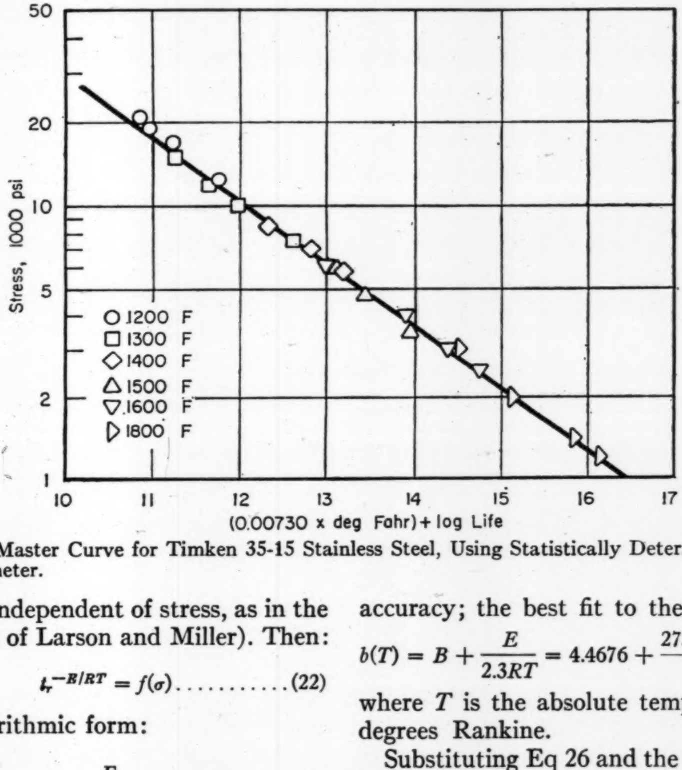


FIG. 5.—Master Curve for Timken 35-15 Stainless Steel, Using Statistically Determined Correlating Parameter.

where A and B are constants, independent of stress and temperature. Equation 25 differs from Eq 5 only in expressing the term $b(T)$ as a linear function of the reciprocal of the absolute temperature rather than as a linear function of temperature. Actually, the values for $b(T)$ can be expressed as the former type of function with only a slight loss of accuracy; the best fit to the data is:

$$b(T) = B + \frac{E}{2.3RT} = 4.4676 + \frac{27318.7}{T} \quad \ldots (26)$$

where T is the absolute temperature in degrees Rankine.

Substituting Eq 26 and the value $A = -4.3500$ into Eq 25 gives:

$$\log t_r = -4.3500 \log \sigma + 4.4676 + \frac{27318.7}{T(^\circ\text{R})} \quad \ldots (27)$$

as the general equation for Timken 35-15 steel. This equation is plotted with the data points in Fig. 6 and is seen to give a good correlation.

The correlating parameter is, accordingly:

$$\log t_r - \frac{27318.7}{T(^\circ\text{R})} = \text{constant at constant stress} \quad \ldots (28)$$

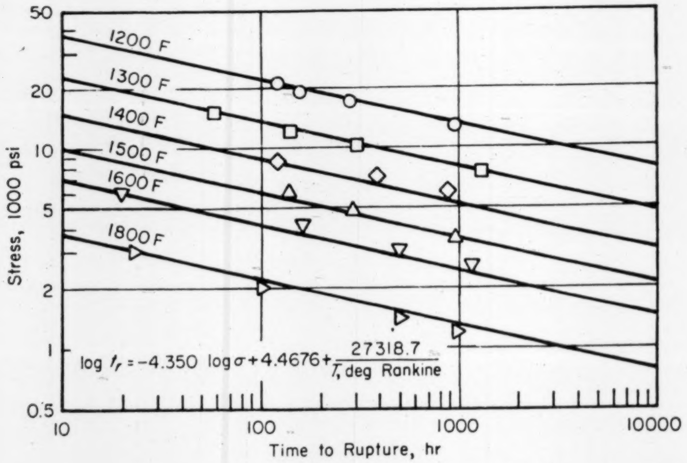


FIG. 6.—Stress-Rupture Plot for Timken 35-15 Stainless Steel, Using Reciprocal of Absolute Temperature in Statistical Equation.

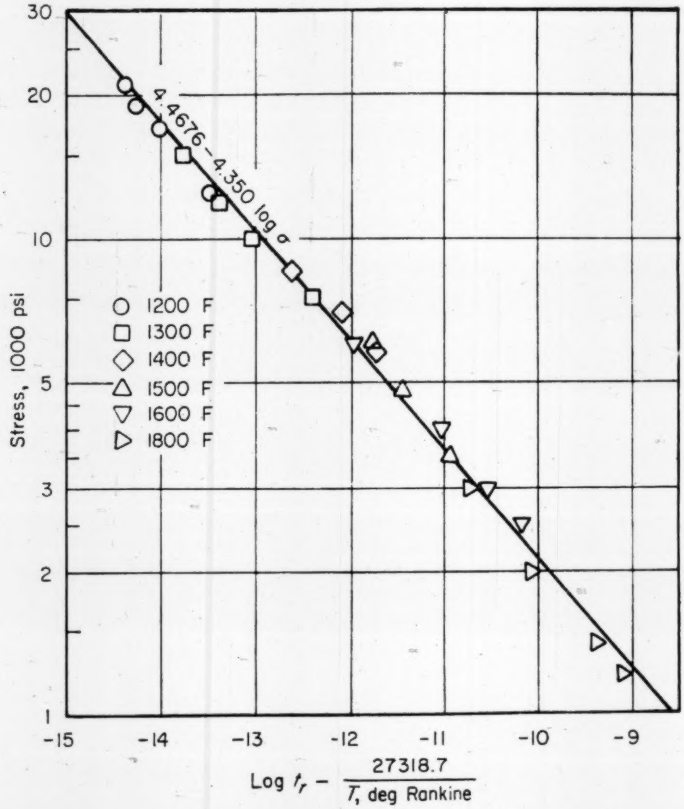


FIG. 7.—Correlation of Data for Timken 35-15 Stainless Steel, Using Method of Reference (5).

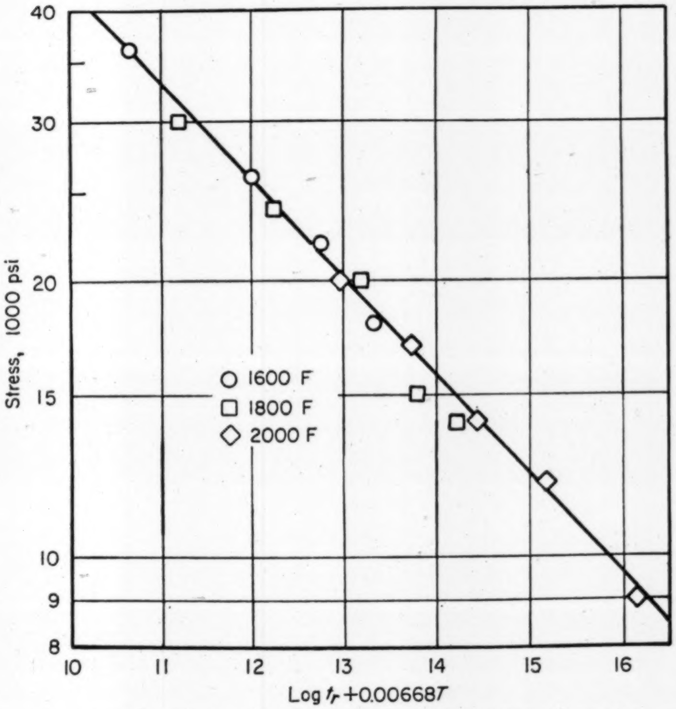


FIG. 8.—Master Curve for Molybdenum (data of Reference (6)).

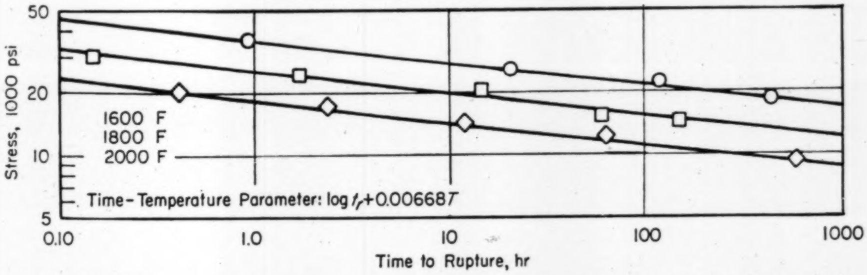


FIG. 9.—Stress-Rupture Plot for Molybdenum, Based on Master Curve (data of Reference (6)).

which is the Orr-Sherby-Dorn parameter in logarithmic form. Figure 7 is a plot of the data correlated by this parameter, and the parameter is seen to give a very good correlation. This is the result of not restricting the basic equation in the manner done by Larson and Miller.

A second example of class I behavior is provided by high-purity molybdenum (6). Figure 8 shows the excellent correlation of the data provided by the parameter ($\log t_r + 0.00668\ T$), and Fig. 9 is the corresponding conventional plot.

Class II:

A more complicated behavior is encountered when the isothermal lines of log stress versus log time-to-rupture are curved rather than straight. This curvature can be accounted for by writing:

$$\log t_r = a(\sigma) \log \sigma + b(T) \ldots\ldots (29)$$

where the coefficient $a(\sigma)$ is a function of

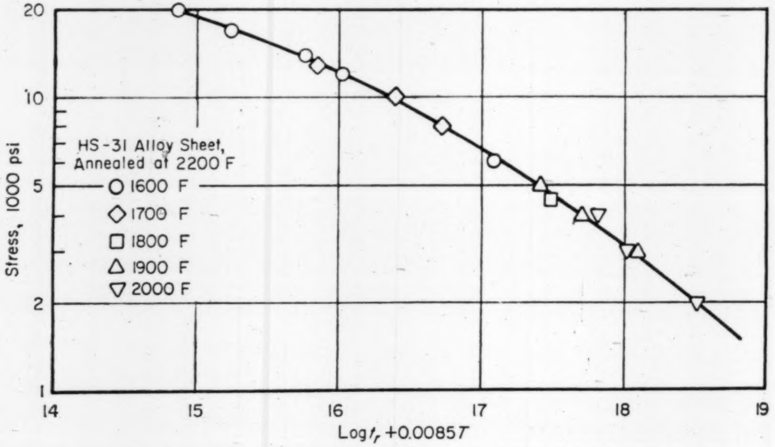


FIG. 10.—Master Curve for Annealed HS-31 Alloy Sheet.

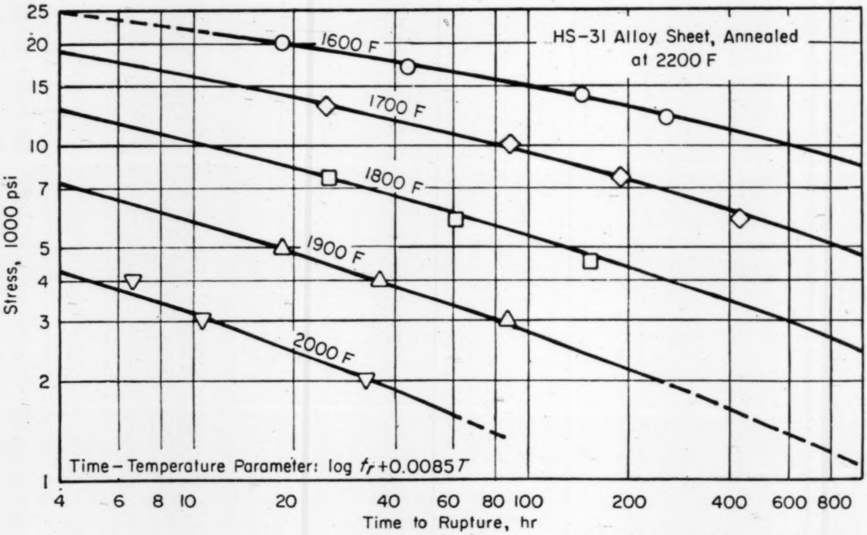


FIG. 11.—Stress-Rupture Plot for Annealed AS-31 Alloy Sheet, Based on Master Curve.

stress. Although the mathematical expression for Eq 29 may be difficult to evaluate, the correlating parameter at constant stress is no more complex than that for Timken 35-15 steel provided that the isothermal curves are parallel and equispaced from one another for equal temperature differences. This condition results in a constant value for the differential $\left(\frac{\partial \psi}{\partial T}\right)_\sigma$ that is independent of stress and temperature. The curvature in the isothermal curves of log stress versus log time-to-rupture causes the master curve to be curved also. Examples of this behavior are provided by the alloys HS-31 (annealed sheet (7)) and A-286 steel (heat-treated bar stock (8)), as

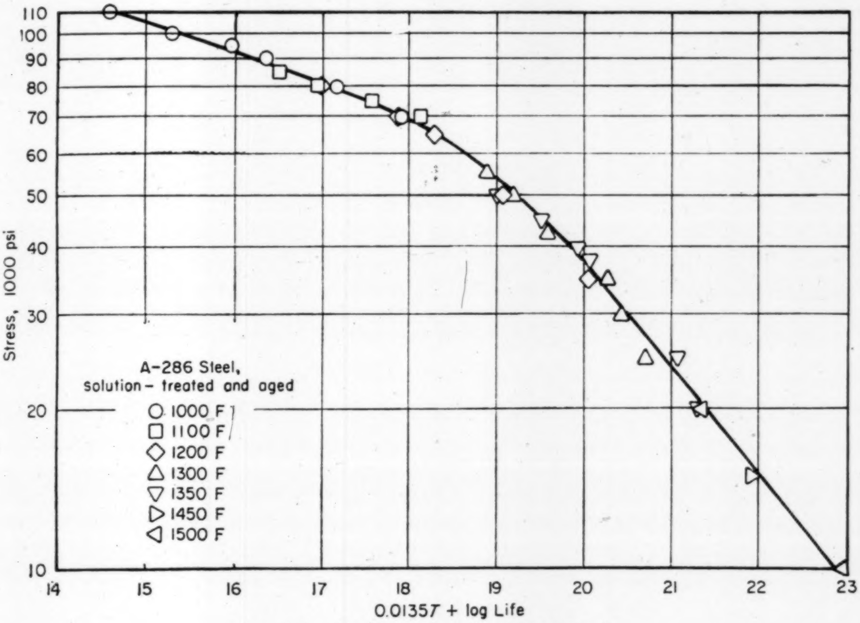


FIG. 12.—Master Curve for A-286 Steel.

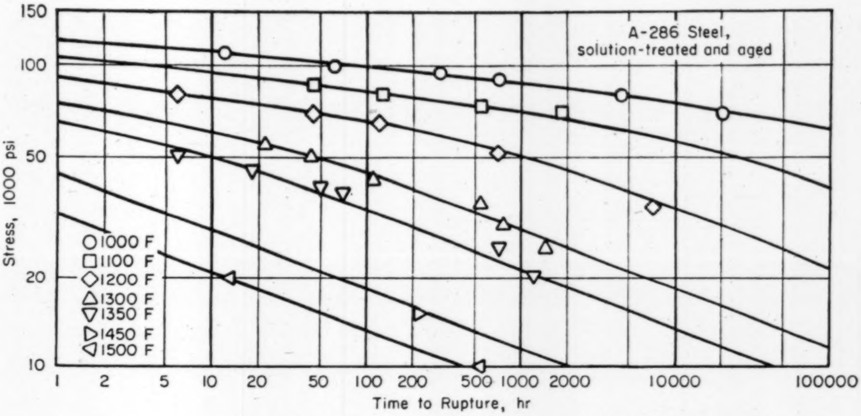


FIG. 13.—Stress-Rupture Plot for A-286 Steel, Based on Master Curve.

shown in Figs. 10 to 13. Inconel 700, which has been studied by Manson and Succop (9), is a further example of class II behavior. The constants in the correlating parameters can be evaluated analytically from the raw data or by scaling of the separation between isothermal lines of log stress versus log time-to-rupture.

Class III:

The isothermal lines of log stress versus log time-to-rupture are straight, as in class I, but they "fan out" so that the slopes of the isothermal lines decrease (slopes are negative) as the temperature increases. Hence, the general form of the function for this behavior is:

$$\log t_r = a(T) \log \sigma + b(T) \ldots\ldots (30)$$

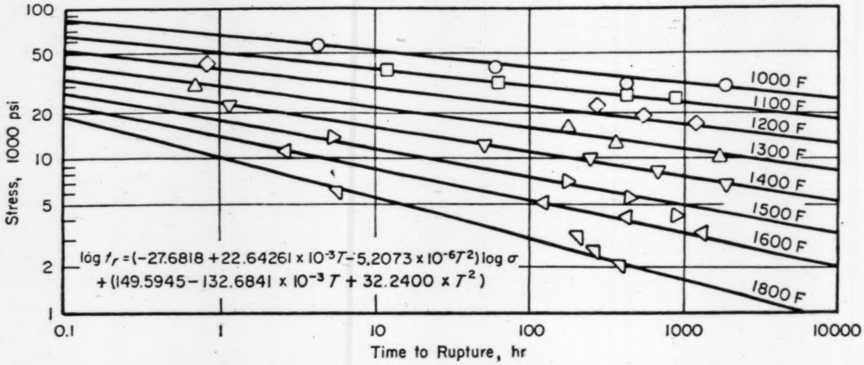


FIG. 14.—Stress-Rupture Plot for Timken 25-20 Stainless Steel, Using Statistically Determined Equation.

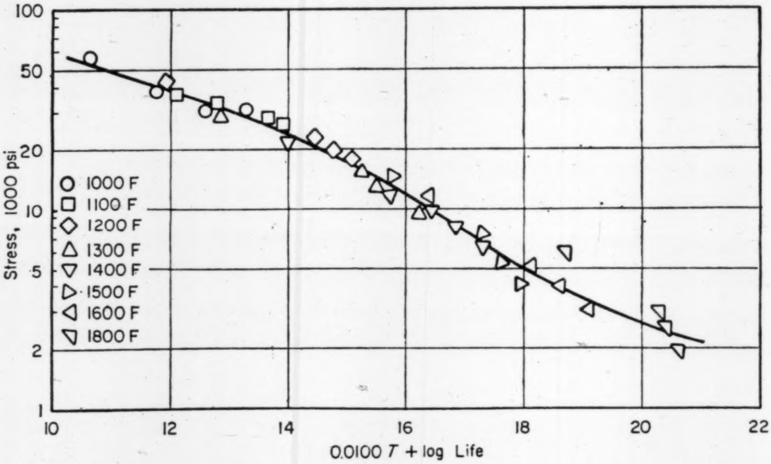


FIG. 15.—Master Curve for Timken 25-20 Stainless Steel, Using Approximate Correlating Parameter.

where the factors $a(T)$ and $b(T)$ are both functions of temperature but not stress.

Timken 25-20 stainless steel provides an example of this behavior. A plot of log stress versus log time-to-rupture for lines of constant temperature is given on page 168 of the Timken Digest (2). Statistical analysis of the raw data for these data yields the equation:

$$\begin{aligned}\log t_r \\ = -(27.6818 &- 22.64261 \times 10^{-3} T \\ &+ 5.20734 \times 10^{-6} T^2) \log \sigma \\ + (149.5945 &- 132.68411 \times 10^{-3} T \\ &+ 32.23997 \times 10^{-6} T^2) \ldots (31)\end{aligned}$$

which involves calculating six constants for Eq 30. The isothermal lines for temperatures from 1000 to 1800 F have been computed from this equation and are plotted in Fig. 14. For our purposes of analysis, the accuracy of Eq 31 is sufficient.

Two points that can now be quickly

disposed of are the accuracy of the parameters ($\log t_r + 0.010\,T$) and the Larson-Miller parameter in correlating the data.

Figure 15 presents the data correlated by the first parameter and Fig 16 by the second. The same detailed remarks made in connection with Timken 35-15 stainless steel may be repeated here: The points for each temperature fall along individual lines, the "average" line is a line of constant life equal to the median life of the data, the scatter of the data about the "average" line is not random but shows definite trends, and gross errors can result from extrapolating short-time lives to predict long-time lives. The degree of correlation shown in Fig. 15 is better than in Fig. 16, so that there is little need to use the more complicated form of the Larson-Miller parameter in place of the factor ($\log t_r + 0.010\,T$) if one's purposes are satisfied with this degree of correlation. Either of these parameters can be improved, however, by expressing the constants as functions of stress and temperature, as well as material.

Manson and Haferd (10) have accomplished this partially with the following time-temperature parameter:

$$\frac{T - T_a}{\log t_r - \log t_a} = \text{constant at constant stress} \ldots (32)$$

where T_a and $\log t_a$ are material constants. This parameter is based on the approximate linearity which they observed in lines of $\log t_r$ against T for constant stress, and on the extrapolation of these lines of constant stress to a conmon point of intersection ($\log t_a$, T_a).

In the case of Timken 25-20 stainless steel, the common point of intersection of the lines of constant stress is reported by Manson and Haferd to be at $\log t_r = 14$, and $T = 100$ F. The correlation of the data by their parameter is shown in Fig. 17. Manson and Haferd eliminated short-time data of less than 30 hr from the scope of their correlation, and these points lie at some distance above the single curve drawn in Fig. 17.

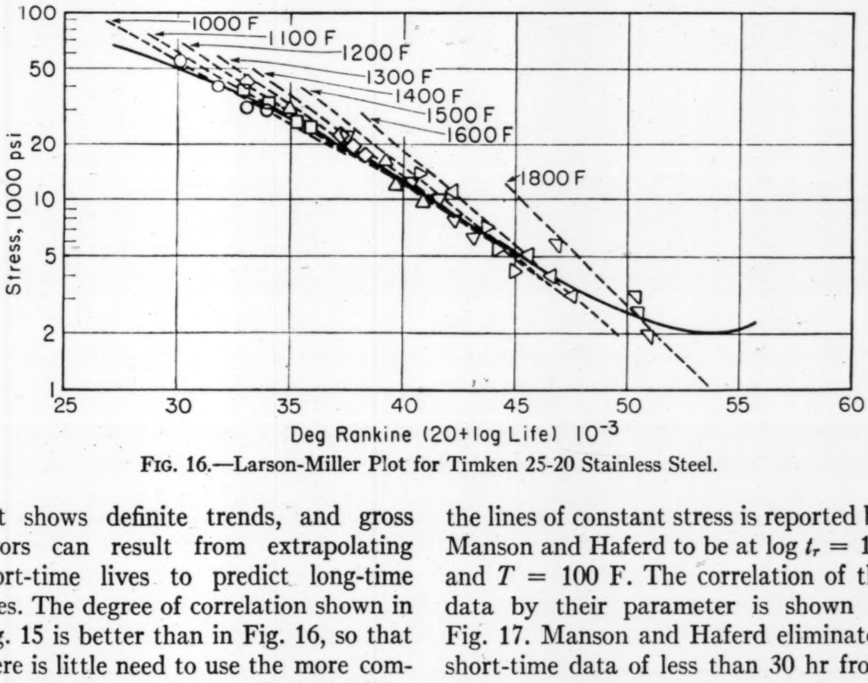


FIG. 16.—Larson-Miller Plot for Timken 25-20 Stainless Steel.

In order to analyze the Manson-Haferd parameter, it is helpful to invert it and study its reciprocal, which is:

$$\frac{\log t_r - \log t_A}{T - T_a} = \text{constant at constant stress} \ldots (33)$$

In this form, one can readily recognize the parameter as $\frac{\Delta \log t_r}{\Delta T}$, or as the incremental form of the differential $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$, which is only part of the general form of the correlating parameter, Eq 14. One should also observe that the values of the Manson-Haferd parameter range from about -60 to -160 for the range of data, and that the reciprocals of these are -0.0167 and -0.0062, respectively; these values are of the order of magnitude of -0.010, which has already been pointed out to be an approximate value satisfied by many high-temperature steels and alloys. Thus, despite the apparent differences between the approaches taken by Manson and Haferd and by Larson and Miller, there is a close similarity in their results.

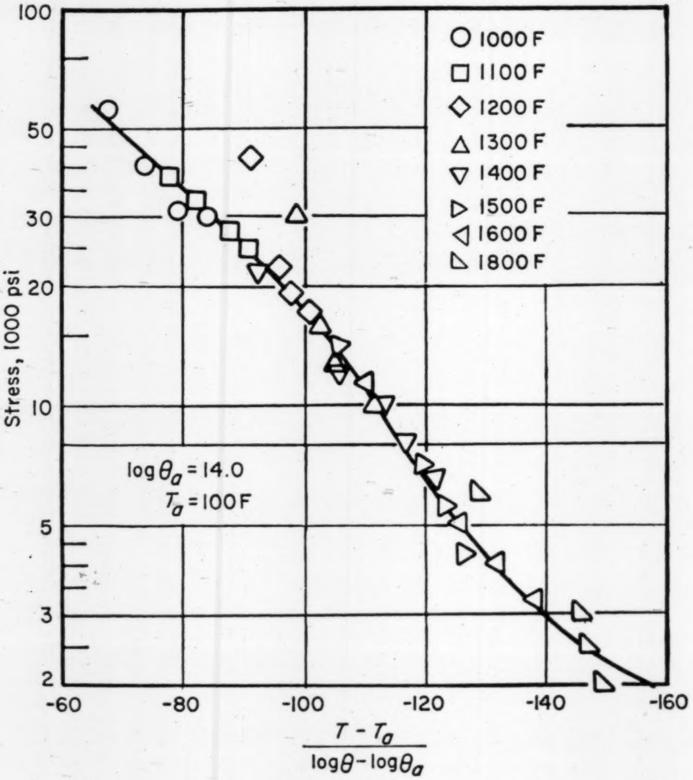


FIG. 17.—Manson-Haferd Plot for Timken 25-20 Stainless Steel.

Although the Manson-Haferd parameter generally gives a better correlation than the Larson-Miller parameter, there is still room for improvement. Part of this improvement must come from realizing that when $\log t_r$ is plotted against either temperature or the reciprocal of the absolute temperature, the lines of constant stress are not generally straight, nor do they extrapolate to a common point of intersection.

These deviations from the ideal behavior assumed by Manson and Haferd require that the factor $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$ in the general correlating parameter, Eq 14, be a function of temperature as well as stress and material. This is observed in the following correlating parameter for Timken 25-20 stainless steel:

$$\begin{aligned}&\log t_r\\&\quad+[(-22.6426\times10^{-3}\\&\qquad\qquad+5.2073\times10^{-6}T)\log\sigma\\&\quad+(132.6841\times10^{-3}\\&\qquad\qquad-32.2400\times10^{-6}T)]\,T\\&\quad=\text{constant at constant stress}\ldots\ldots\ldots\ldots(34)\end{aligned}$$

where T is the temperature in degrees Fahrenheit. This parameter is readily derived from the empirical Eq 31 that was developed for this steel.[3] The data are correlated by this parameter in Fig. 18. Since the isothermal lines of log σ against log t_r approximated straight lines on the conventional plot, Fig. 14, the plot of log σ against the parameter of Eq 34 is a straight line. Note how well the data over the entire range agree with this line.

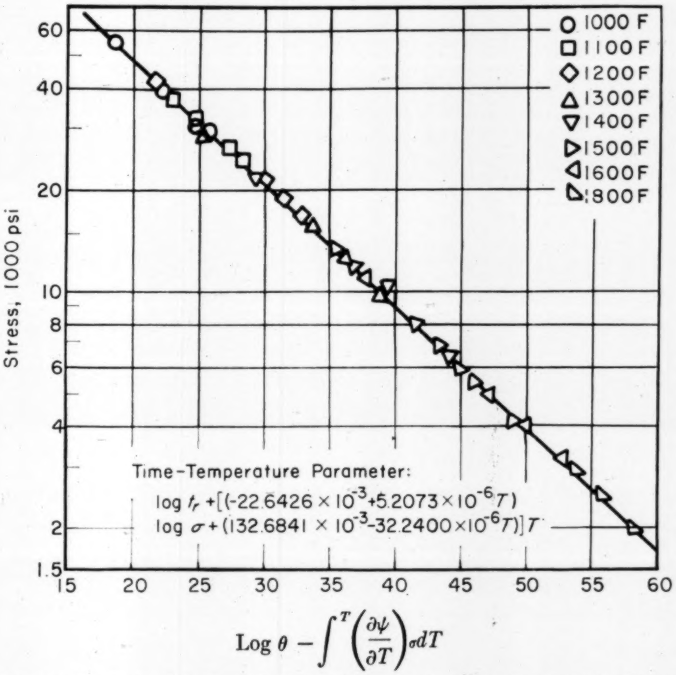


FIG. 18.—Master Curve for Timken 25-20 Stainless Steel, Using Statistically Determined Correlating Parameter.

To apply the Orr-Sherby-Dorn correlation to Timken 25-20 steel both factors $a(T)$ and $b(T)$ in Eq 30 are assumed to be linear functions of the reciprocal of the absolute temperature. Statistical analysis of the data then gives:

$$\log t_r=\left(8.2344-\frac{26934}{T(^\circ\mathrm{R})}\right)\log\sigma$$

$$+\left(-48.9033+\frac{142342}{T(^\circ\mathrm{R})}\right)\ldots.(35)$$

Figure 19 shows the isothermals calculated from this equation along with the data points. Although the agreement is not so good as that provided by Eq 31, Eq 35 has the advantage of having fewer constants. It is sufficiently accurate for our purposes here.

---

[3] Alternatively, the four coefficients in Eq 34 could have been evaluated by making a preliminary plot of the data in the form of Fig. 14, measuring the separation between isothermal lines for four conditions at different stresses and temperatures, and evaluating the constants in a set of four simultaneous equations. This is much easier than deriving Eq 31 first and then differentiating it.

Differentiating Eq 35 with respect to temperature gives:

$$\left(\frac{\partial \log t_r}{\partial T}\right)_\sigma = (-26934.0 \log \sigma + 142342) \frac{\partial}{\partial T}\left(\frac{1}{T}\right) \quad \text{..(36)}$$

Note that this last equation indicates that the correlating parameter must include a stress factor.

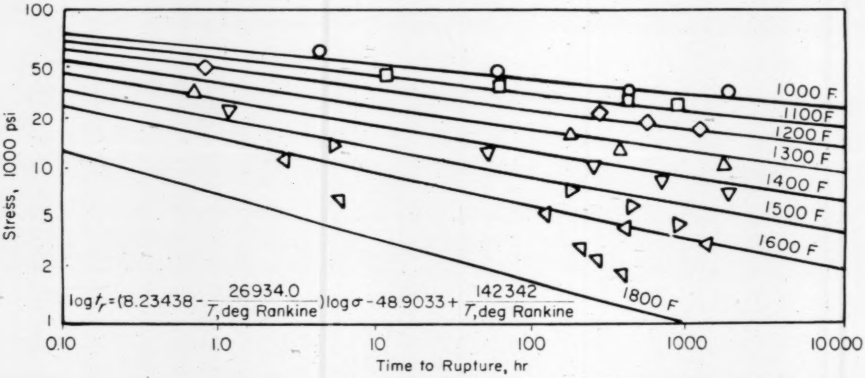


FIG. 19.—Stress-Rupture Plot for Timken 25-20, Using Reciprocal of Absolute Temperature in Statistical Equation.

To obtain a correlation of the type proposed by Orr, Sherby, and Dorn, one can use a convenient dodge. This dodge consists in making an approximation and then plotting in a manner that minimizes the error introduced by the approximation. To do this, one first evaluates the differential $\left(\frac{\partial \log t_r}{\partial T}\right)_\sigma$ at an average stress, say, 10,000 psi, so that Eq 36 becomes:

$$\left(\frac{\partial \log t_r}{\partial T}\right)_{\text{avg}} = 34606 \frac{\partial}{\partial T}\left(\frac{1}{T}\right) \quad \text{....(37)}$$

and the correlating parameter becomes:

$$\log t_r - \frac{34606}{T(°\text{R})} = \text{constant at constant stress} \quad \text{..(38)}$$

The relation between this parameter and the stress can now be allowed to be defined graphically by the data points, as in Fig. 20, which shows a very good correlation of the data. The error involved in the approximation distorts the relation from the straight line obtained in Fig. 18 with the more exact parameter to the curved line in this last figure. Over the range of variables, the correlation in Fig. 20 has lost little accuracy over that in Fig. 18; average values are approximated very well, while extremes suffer slightly. As the correlation is extrapolated more and more, the errors become larger. If the isothermals in Fig. 19 were replotted to agree with Fig. 20, they would also show a slight curvature and would indicate a better correlation than that given by Eq 35.

The important point to be made here is not that the Orr-Sherby-Dorn correlation is inaccurate. On the contrary, it can be extremely accurate and useful over rather wide ranges and has much practical utility. Of more importance to the theorist is the fact that the correlation has a "built-in" compensation that

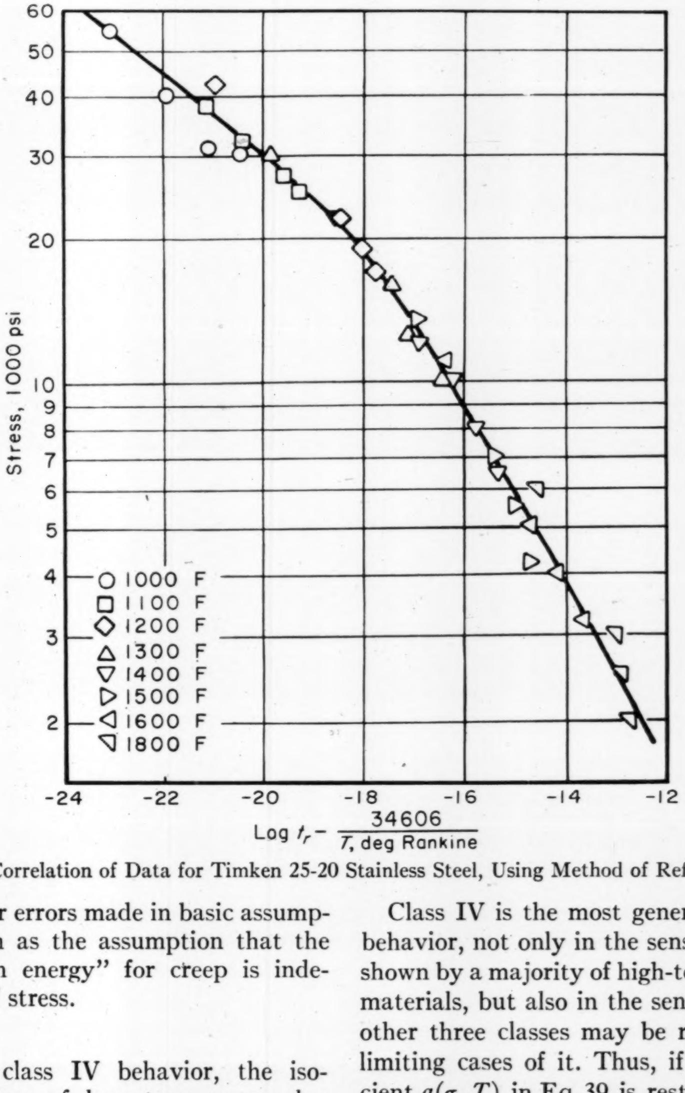


FIG. 20.—Correlation of Data for Timken 25-20 Stainless Steel, Using Method of Reference (5).

corrects for errors made in basic assumptions, such as the assumption that the "activation energy" for creep is independent of stress.

Class IV:

In the class IV behavior, the isothermal lines of log stress versus log time-to-rupture are curved, the curvature being downwards and increasing as the temperature increases, so that the curves "fan out." These features can be expressed by the equation:

$$\log t_r = a(\sigma, T) \log \sigma + b(T) \quad \text{.....(39)}$$

where $a(\sigma, T)$ is a function of both temperature and stress and $b(T)$ is a function of temperature only.

Class IV is the most general type of behavior, not only in the sense that it is shown by a majority of high-temperature materials, but also in the sense that the other three classes may be regarded as limiting cases of it. Thus, if the coefficient $a(\sigma, T)$ in Eq 39 is restricted to a constant value, class I behavior is obtained; if the coefficient is restricted to a function of stress only, class II behavior is obtained; and if the coefficient is restricted to a function of temperature only, class III behavior is obtained.

The alloy S-590 provides an example of class IV behavior (12). The empirical equation derived statistically to approximate the data for S-590 alloy is:

$$\log t_r = (-54.98878 + 35.4818 \times 10^{-3}T - 7.20513 \times 10^{-6}T^2)\left(\log\sigma + \frac{10}{\log\sigma}\right) + (404.002881 - 272.505 \times 10^{-3}T + 56.0224 \times 10^{-6}T^2) \text{ for } \sigma > 1500 \text{ psi} \ldots (40)$$

The author emphasizes that the empirical equation has been derived for purposes of analysis only. In order to fit the data with a high degree of accuracy, a large number of constants had to be evaluated, and the equation should not be applied to stresses below 1500 psi. The accuracy of the equation can be judged from its agreement with the data points, as shown in Fig. 21. Note that the data are represented by a continuous function, or by smooth curves, and that it is unnecessary to assume the existence of short straight-line segments and sharp breaks, as in the correlation of Grant and Bucklin (**12**).

The use of a single, smooth, continuous function to represent these data is further demonstrated in Fig. 22, where these data have been reduced to a single curve by the general correlating parameter, Eq 14, where:

$$\int^T \left(\frac{\partial\psi}{\partial T}\right)_\sigma dT = (35.4818 \times 10^{-3}T - 7.20513 \times 10^{-6}T^2)\left(\log\sigma + \frac{10}{\log\sigma}\right) - (272.505 \times 10^{-3} - 56.0224 \times 10^{-6}T^2) \ldots (41)$$

The terms in this parameter were determined from the empirical formula (Eq 40) derived from the raw stress-rupture data, and the parameter is seen to contain two less constants than the complete function for log t_r. Obviously, if one were interested only in the parameter, it would have been unnecessary to have derived the complete function first. The parameter can also be determined directly from the raw data, and, since it contains fewer constants, this is an easier task than determining the complete function. The results of this analysis emphasize that it is not necessary to assume the existence of breaks in the behavior in order to correlate stress-rupture data.

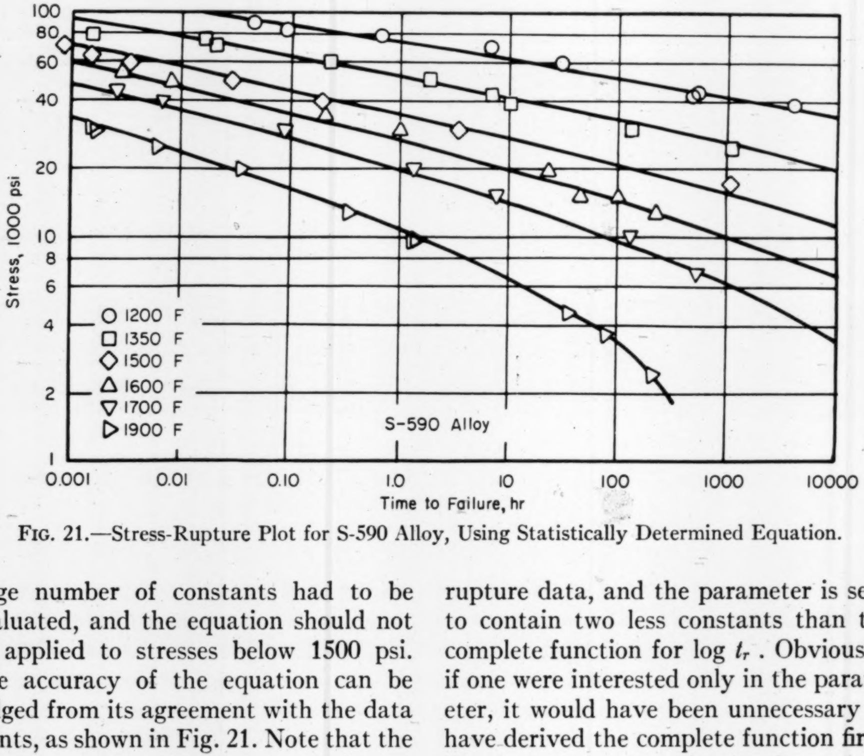


FIG. 21.—Stress-Rupture Plot for S-590 Alloy, Using Statistically Determined Equation.

CONCLUSIONS

1. The general form of the parameter for correlating stress-rupture behavior at constant stress can be expressed as:

$$\psi - \int^{T} \left(\frac{\partial \psi}{\partial T}\right)_{\sigma} dT = \text{constant} \quad \text{....(14)}$$

where:

$$\psi = \log t_r$$

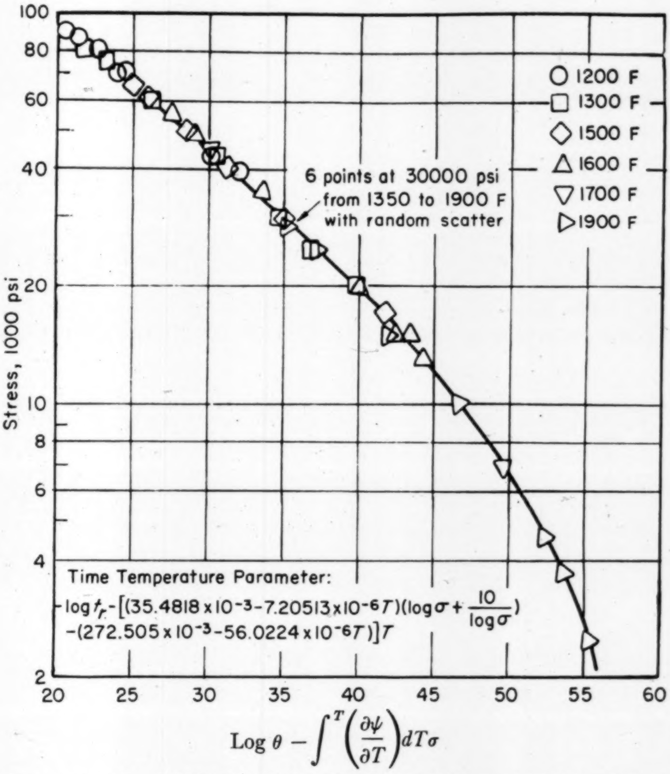


FIG. 22.—Master Curve for S-590 Alloy, Using Statistically Determined Correlating Parameter.

2. The differential $\left(\frac{T\partial}{\psi\partial}\right)_{\sigma}$ corresponds to the separation between isothermal lines of $\log \sigma$ versus $\log t_r$ on the conventional plot of stress-rupture data.

3. In general, $\left(\frac{\partial \psi}{\partial T}\right)_{\sigma}$ is a function of stress, σ, and temperature, T, as well as material.

4. As an approximation only, $\left(\frac{\partial \psi}{\partial T}\right)_{\sigma}$ can be assigned a constant value equal to -0.010 per deg Fahr, so that the correlating parameter becomes:

$$\log t_r + 0.010T = \text{constant at constant stress} \quad \text{..(15)}$$

where T is in degrees Fahrenheit. This parameter is simpler to use than the Larson-Miller parameter and gives about the same degree of correlation. Its use should be confined to cases where it is desired to compare the behavior of several materials over similar ranges of rupture life. Neither it nor the Larson-Miller should be used for extrapolation or where a high degree of accuracy is needed.

5. A higher degree of accuracy over

the parameter in item 4 can be obtained by evaluating $\left(\frac{\partial\psi}{\partial T}\right)_\sigma$ as a material constant, so that:

$$\log t_r - CT = \text{constant at constant stress} \quad (42)$$

where the material constant, C, can be determined analytically from the raw data or by measuring the separation between isothermal curves of log stress versus log time-to-rupture. In the same manner, the Larson-Miller parameter can be improved, as has long been recognized, by evaluating the constant in it for each material. This improvement in accuracy is not without some sacrifice in utility. Because different materials will have different values of C in their parameters, it will not be possible to compare their behaviors by plotting the data for them on the same scale and on the same graph. Since the separation between isothermal curves of log stress versus log time-to-rupture generally varies with the stress and temperature, the parameter generally cannot be used to extrapolate the data over wide ranges. Best results are obtained when the constant C is evaluated at the average conditions. The correlation is exact only when the conventional plot of log stress versus log time-to-rupture gives a series of isothermal lines that are parallel and equally spaced from one another for equal temperature differences.

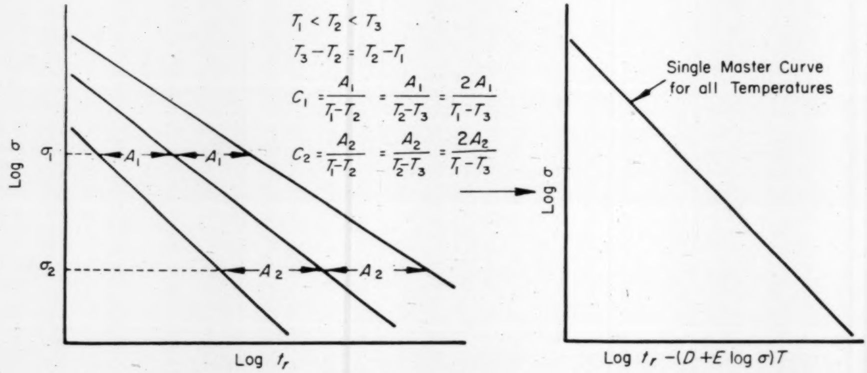

$T_1 < T_2 < T_3$
$T_3 - T_2 = T_2 - T_1$
$C_1 = \frac{A_1}{T_1-T_2} = \frac{A_1}{T_2-T_3} = \frac{2A_1}{T_1-T_3}$
$C_2 = \frac{A_2}{T_1-T_2} = \frac{A_2}{T_2-T_3} = \frac{2A_2}{T_1-T_3}$
σ_1
σ_2
A_1
A_1
A_2
A_2
Log σ
Log t_r
Single Master Curve for all Temperatures
Log σ
Log t_r −(D + E log σ)T

FIG. 23.—Schematic Representation for Derivation of Correlating Parameter.

6. Where a still higher degree of accuracy is required, or the behavior is more complex, it may be necessary to evaluate the differential as a function of stress for the material under consideration; that is, the parameter becomes:

$$\log t_r - C(\sigma)\,T = \text{constant} \quad (43)$$

If the isothermal curves "fan out" in such a way that the amount of separation at constant stress varies only with the stress and not with the temperature, then the amount of separation must be measured at several stress levels in order to obtain an accurate value of the parameter. If the isothermal curves are straight lines, then the amount of separation is necessarily a linear function of log σ—that is $C = D + E \log \sigma$. If the amounts of separation are A_1 at σ_1 for a temperature difference ΔT_1 and A_2 at a different stress σ_2 and a temperature difference ΔT_2, then $C_1 = \frac{A_1}{\Delta T_1}$ and $C_2 = \frac{A_2}{\Delta T_2}$ and the constants D and E can be evaluated from the two simultaneous equations:

$$C_1 = D + E \log \sigma$$
$$C_2 = D + E \log \sigma_2$$

Then the correlating parameter is $[\log t_r - (D + E \log \sigma)\, T]$. Figure 23 illustrates this schematically. If the isothermal lines are curved but are separated from each other by an amount which varies linearly with log σ, and if the separation remains independent of temperature, then the same type of parameter can be used but the master curve will show a slight curvature rather than being straight. The type of behavior described here is one that will give straight lines of constant stress when log t_r is plotted against T. If it can be further assumed that these straight lines project to a common point of intersection, the Manson-Haferd type of parameter, also involving two constants, can be used with a good degree of correlation.

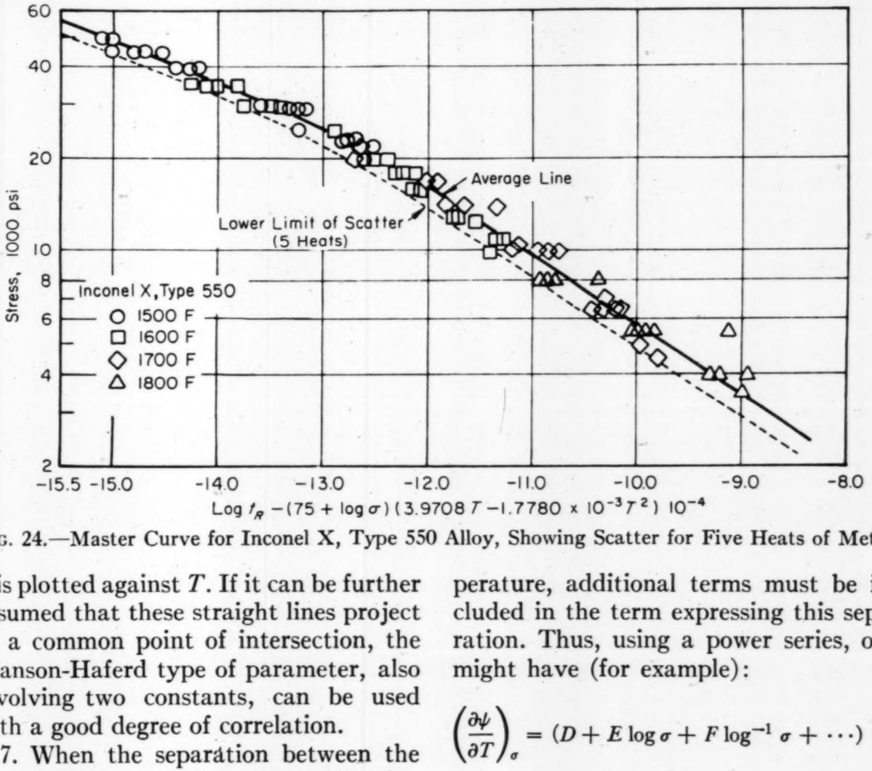


FIG. 24.—Master Curve for Inconel X, Type 550 Alloy, Showing Scatter for Five Heats of Metal.

7. When the separation between the isothermal lines of log stress versus log time-to-rupture varies with the temperature as well as the stress, this variation must be included in the parameter in order to obtain a high degree of accuracy. The parameter will then have the form:

$$\log t_r - C(\sigma, T)\, T = \text{constant} \quad \ldots\ldots (44)$$

If the separation is linear with respect to log σ and T, or approximately so, the constants in an expression such as $(C_1 + C_2 \log \sigma)(C_3 + C_4 T)$, or an equivalent expression, must be evaluated. Figure 24 shows the correlation obtained by analyzing stress-rupture results from five heats of Inconel 550 alloy in this manner **(15)**.

8. As the stress-rupture behavior becomes more complex so that the separation of the isothermals is no longer linear with the log of the stress and with temperature, additional terms must be included in the term expressing this separation. Thus, using a power series, one might have (for example):

$$\left(\frac{\partial \psi}{\partial T}\right)_\sigma = (D + E \log \sigma + F \log^{-1} \sigma + \cdots)$$
$$(G + HT + JT^2 + \cdots) \ldots\ldots (45)$$

and can include as many terms as necessary in order to obtain the desired degree of accuracy. Using the reciprocal of the absolute temperature, as suggested in the correlation of Dorn et al **(16)**, may give better results or a simpler expression in some cases.

9. Adding more and more terms to a correlation will obviously improve its accuracy. Where a high degree of accuracy is required, the added work this entails is justified. At other times, high accuracy is not essential or the correlation is to be applied only over a limited range of variables. In those cases, a simpler correlation can be just as useful and, over a limited range, just as accurate as a more complex one. Of course, the final accuracy of the correlation will be limited by the scatter in the original data.

10. The use of correlations to justify theories of creep-rupture must be made with caution. A careful analysis often reveals that apparently random scatter is really a systematic error. Statistical procedures that eliminate personal bias are recommended, and graphical methods that obscure mistakes in theory are to be avoided. This precaution is not so necessary when only a correlation is desired and the theory is not intended to be tested rigorously.

REFERENCES AND BIBLIOGRAPHY

(1) C. W. Phillips and M. J. Sinnott, "A Statistical Study of the Stress-Rupture Test," *Transactions*, Am. Soc. Metals, Vol. 46, pp. 63–86 (1954).

(2) Digest of Steels for High-Temperature Service, 5th edition, Timken Roller Bearing Co., Steel and Tube Division, Canton, Ohio (1946).

(3) Keith J. Laidler, Chemical Kinetics, McGraw-Hill, New York, N. Y. (1950).

(4) F. R. Larson and J. Miller, "A Time-Temperature Relationship for Rupture and Creep Stresses," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 74, July 1952, pp. 765–771.

(5) R. L. Orr, O. D. Sherby, and J. E. Dorn, "Correlations of Rupture Data for Metals at Elevated Temperature," *Transactions*, Am. Soc. Metals, Vol. 46, pp. 113–128 (1954).

(6) J. W. Pugh, "The Tensile Properties of Molybdenum at Elevated Temperatures," *Transactions*, Am. Soc. Metals, Vol. 47, pp. 984–1001 (1955).

(7) J. W. Hoffman and L. F. Ecker, "Third Progress Report on the Investigation of Materials for Turbo-Jet Engine Tail Pipes," Report No. NA-48-194, Engineering Research Laboratory, North American Aviation, Inc., Los Angeles, Calif., Feb. 27, 1948.

(8) C. B. Craver, G. N. Aggen, and W. W. Dyrkacz, "A Study of Precipitation-Hardening of A-286 Alloy. Part I.—Identification of Microconstituents and Their Correlation with Hardness," Allegheny Ludlum Steel Corp., Research Laboratories, Watervliet, N. Y., Report H-0111-No. 8, March 1, 1955.

(9) S. S. Manson and G. Succop, "Stress-Rupture Properties of Inconel 700 and Correlation on the Basis of Several Time-Temperature Parameters," Symposium on Metallic Materials for Service at Temperatures Above 1600 F, Am. Soc. Testing Mats., p. 40 (1955). (Issued as separate publication *ASTM STP No. 174.*)

(10) S. S. Manson and A. M. Haferd, "A Linear Time-Temperature Relation for Extrapolation of Creep and Stress-Rupture Data, Natl. Advisory Com. Aeronautics, Technical Note 2890 (1953).

(11) E. S. Machlin and A. S. Nowick, "Stress Rupture of Heat-Resisting Alloys as a Rate Process," Natl. Advisory Com. Aeronautics, Technical Note 1126, Sept. 1946.

(12) Nicholas J. Grant and Albert G. Bucklin "On the Extrapolation of Short-Time Stress-Rupture Data," *Transactions*, Am Soc. Metals, Vol. 42, pp. 720–751 (1950).

(13) C. L. Clark, A. E. White, and G. J. Guarnieri, "A New Application for the Short-Time High Temperature Tensile Test," *Transactions*, Am. Soc. Metals, Vol. 26, p. 1035 (1938).

(14) R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs., Vol. 135, p. 559 (1939).

(15) Creep Rupture Test Data, Inconel X, Type 550, Inco Current Data Report No. 1, International Nickel Co., Ltd., Nov. 1952.

(16) J. E. Dorn, P. Pietrokowsky, and T. E. Tietz, "The Effect of Alloying Elements on the Plastic Properties of Aluminum Alloys," *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs., Vol. 188, pp. 933–943 (1950); *Journal of Metals*, July 1950.

(17) A. T. Robinson, T. E. Tietz, and J. E. Dorn, "The Functions of Alloying Elements in the Creep Resistance of Alpha Solid Solutions of Aluminum," *Transactions*, Am. Soc. Metals, Vol. 44, pp. 896–928 (1952).

(18) O. D. Sherby, R. A. Anderson, and J. E. Dorn, "The Effect of Alloying Elements on the Elevated Temperature Plastic Properties of Alpha Solid Solutions of Aluminum," *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs. Vol. 191, pp. 643–652 (1951); *Journal of Metals*, Aug. 1951; Discussion, *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs., Vol. 194, p. 529 (1952).

(19) O. D. Sherby and J. E. Dorn, "Creep Correlations in Alpha Solid Solutions of Aluminum," *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs., Vol. 194, pp. 959–964 (1952); *Journal of Metals*, Sept. 1952.

(20) Oleg D. Sherby and John E. Dorn, "Some Observations on Correlations Between the Creep Behavior and the Resulting Structures in Alpha Solid Solutions," *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs., Vol. 197, pp. 324–330 (1953); *Journal of Metals*, Feb. 1953.

(21) C. Dean Starr, E. C. Vicars, A. Goldberg, and J. E. Dorn, "Effect of Alloying Elements on Grain Boundary Relaxation in Alpha Solid Solutions of Aluminum," *Transactions*, Am. Soc. Metals, Vol. 45, pp. 275–285 (1953).

(22) Oleg D. Sherby, Raymond L. Orr, and John E. Dorn, "Creep Correlations of Metals at Elevated Temperatures," *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs., Vol. 200, pp. 71–80, 1959); *Journal of Metals*, Jan. 1954.

(23) Oleg D. Sherby, R. Frenkel, J. Nadeau, and John E. Dorn, "Effect of Stress on the Creep Rates of Polycrystalline Aluminum Alloys Under Constant Structure," *Transactions*, Am. Inst. Mining, Metallurgical, Petroleum Engrs., Vol. 200, pp. 275–279 (1954); *Journal of Metals*, Feb. 1954.

(24) C. Zener and J. H. Hollomon, "Effect of Strain Rate Upon Plastic Flow of Steel," *Journal of Applied Physics*, Vol. 15, pp. 22–32, Jan. 1944.

(25) Nicholas J. Grant and Albert G. Bucklin, "Creep-Rupture and Recrystallization of Monel from 700 to 1700 F," *Transactions*, Am. Soc. Metals, Vol. 45, pp. 151–170 (1953).

(26) A. S. Nowick and E. S. Machlin, "Quantitative Treatment of the Creep of Metals by Dislocation and Rate-Process Theories," Natl. Advisory Com. Aeronautics, Technical Note 1039, April 1946.

(27) F. Garofalo, G. V. Smith, and B. W. Royle, "Validity of Time-Compensated Temperature Parameters for Correlating Creep and Creep-Rupture Data," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 78, Oct. 1956, pp. 1423–1429.

(28) S. S. Manson and W. F. Brown, Jr., "An Investigation of Time-Temperature-Stress Relations for the Correlation and Extrapolation of Stress-Rupture Data," Natl. Advisory Com. Aeronautics, Technical Note (1954).

STRENGTH OF STRUCTURAL ALLOYS UNDER RAPID HEATING AND LOADING*

BY J. R. KATTUS[1] AND D. W. MCDOWELL, JR.[2]

SYNOPSIS

The rapid tensile and short-time creep properties are presented for three commercial stainless steel sheet materials, three commercial nickel-base sheet alloys, and one experimental stainless steel that had been cold rolled at −105 F. The specimens evaluated were heated to test temperatures up to 2000 F within 30 sec. Tensile determinations were made at a strain rate of 0.1 in. per in. per sec; creep properties were determined at tensile stresses that caused rupture in times ranging from 1 min to 30 min. Data of this type are needed for the safe and efficient design of missiles that are subjected to severe loads and elevated temperatures during short service lives of only a few seconds or minutes. The special equipment used to obtain the data is described.

The load-carrying capacity of all of the alloys increases with decreasing exposure times from 30 min to 1 sec at temperature under load. The high-strength stainless steels are stronger than the nickel alloys at the lower temperatures, but the nickel alloys retain useful load-carrying capacity to higher temperatures than the stainless steels do, the short-time creep strengths of the nickel alloys in the range 1400 to 1800 F being roughly equivalent to those of the stainless steels in the range 1000 to 1400 F.

At temperatures up to 1200 F, type 304 stainless steel that had been cold rolled at −105 F to achieve a yield strength exceeding 350,000 psi at room temperature had higher tensile strength than any commercial grade of stainless steel.

Most engineering structures used at elevated temperatures, such as steam power equipment, chemical processing equipment, jet engines, and internal combustion engines, are designed to function for many hours or years. Evaluations of the load-carrying capacity of structural materials for these applications have usually involved creep tests for such extended periods. Many missiles, on the other hand, are designed for a service life of only a few seconds or a few minutes. During this short time, certain structural components are subjected to rapidly applied stresses and elevated temperatures. The load-carrying capacity of the structural materials under these conditions may be considerably different from that determined in conventional tension and creep tests. Consequently, an investigation was made of the tensile and creep strength of several stainless steels and nickel alloys under conditions of rapid heating, rapid loading, and short exposures to high temperature and stress. The results of this work are expected to be useful in the design of missiles.

---

* Presented at the Sixty-third Annual meeting of the Society June 26–July 1, 1960.

[1] Head of Metallurgy Division, Southern Research Institute, Birmingham, Ala.

[2] Metallurgist, Development and Research Division, International Nickel Co., Inc., New York, N. Y.

The purpose of this paper is to illustrate the effects of variations in heating and loading time from 1 sec to 30 min on the load-carrying capacity of three stainless steels and three nickel-base sheet materials at temperatures up to a maximum of 2000 F. A secondary purpose is to illustrate the beneficial effects of cold working at subzero temperatures on the short-time strength of austenitic stainless steel.

EQUIPMENT

The equipment that was developed at Southern Research Institute for the evaluation of the strength of metals under conditions of rapid heating and loading has been described in detail elsewhere (**1, 2, 3**).[3] Two methods were used to apply load to the specimens: (1) with constant strain rate for the tension tests, and (2) with constant tensile load for the creep tests.

For the tension tests, a machine was used with a mechanical screw-driven crosshead and a maximum loading capacity of 7000 lb. With a speed range of 20 to 1, driving power was supplied to the tensile loading frame by a 2-hp power package consisting of a d-c motor and electronic control panel. A flywheel was built into the drive to smooth out the shock of the rapidly applied load. The screw was coupled to the drive through an electromagnetically energized friction clutch. The movable crosshead was started almost instantaneously by engaging the clutch as the flywheel ran at constant speed. Resistance strain gages served as the basic sensing elements in both the extensometers and load cells used in the tension tests. The load cell was linked in series with the specimen, whereas the extensometer, which averaged strain on opposite edges of the specimen, was clipped onto the specimen at the gage points. Excitation was supplied to both the load cell and the extensometer from a stable source of alternating current at a frequency of 5000 cps. The outputs from these transducers were small a-c voltages proportional to load and to strain. During each tension test, these calibrated outputs were amplified, demodulated, filtered, and then fed into the *X* and *Y* axes of an oscilloscope. The stress-strain curve, which was traced on the face of the scope, was photographed with a Polaroid Land camera.

Both the tensile and creep specimens, which had gaged sections $\frac{1}{2}$ in. wide by 2 in. long, were resistance-heated by current supplied through a welding transformer. Temperature control was provided by 36-gage Chromel-Alumel thermocouples flash-welded to the middle of the gage length and by a temperature recorder-controller working in conjuntion with the resistance heating unit. Thermocouples were welded at the gage points for measuring temperature uniformity along the gage length. These thermocouples actuated a termperature recorder that monitored the temperature at the gage points. Since, with resistance heating, the center of the gage section tends to become hotter than the ends, a slight controlled stream of cooling air directed at the center was used to even out the temperature distribution. With these techniques, uniformity and control of the specimen temperature was maintained within ± 1 per cent of the desired test temperature.

For the short-time creep tests, a

---

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

beam mounted on hardened knife edges was used to multiply the applied tensile force from static weights on a pan at the long end of the beam. An extensometer similar to the type used in the tension tests was clipped onto the creep specimens at the gage points. The extensometer was supplied with d-c excitation from a battery. The calibrated output from the extensometer, which was a d-c voltage in the range of 0 to 20 mv, was fed directly into a recording potentiometer, which recorded a strain-time curve for each creep test.

PROCEDURE

For each elevated test temperature, the tension specimens were heated to temperature within 10 sec, held at temperature 10 sec, and then loaded to failure at a strain rate of 0.1 in. per in. per sec. At this strain rate, which is representative of the rapid rates of straining in missile structures, the loading time to failure is about 1 sec. A stress-strain curve was recorded to 2 per cent offset during each tension test. Beyond that point, only load to failure was recorded. The recorded data permitted the determination of modulus of elasticity, ultimate strength, and yield strength at various degrees of offset up to 2 per cent. Two or three tensile determinations were made for each test condition.

At each creep test temperature, specimens of each material were evaluated at a number of constant load levels selected to produce rupture in various lengths of time ranging from 1 min to $\frac{1}{2}$ hr. For each test, the dead-weight load was applied at room temperature. The specimen was heated to test temperature within 30 sec and then held at constant temperature and constant load until it ruptured. Strain was recorded as a function of time both during heating and during the time that the specimen was at constant temperature and constant load. The results of the short-time creep tests showed the load-carrying capacity of structural materials when they supported loads at elevated temperatures for finite periods of time up to $\frac{1}{2}$ hr. This time was representative of the maximum service time for most missiles.

For each test material, the maximum temperature was the same for the tensile and creep evaluations; the minimum temperature for the creep evaluations was, roughly, the temperature below which short-time creep properties are not important in the design of structures that must function within some limited amount of distortion or deformation. At the higher temperatures, short-time creep strength is lower than tensile yield strength and, therefore, must be taken into consideration in the design of structural members that must sustain loads for short periods of time at these temperatures. At lower temperatures, the short-time creep strength exceeds the tensile yield strength; consequently, the initial deformation is excessive when stresses are applied that are sufficient to cause significant creep within $\frac{1}{2}$ hr. Under these conditions, the tensile yield strength at some specified degree of offset is the maximum allowable design stress.

ALLOYS EVALUATED

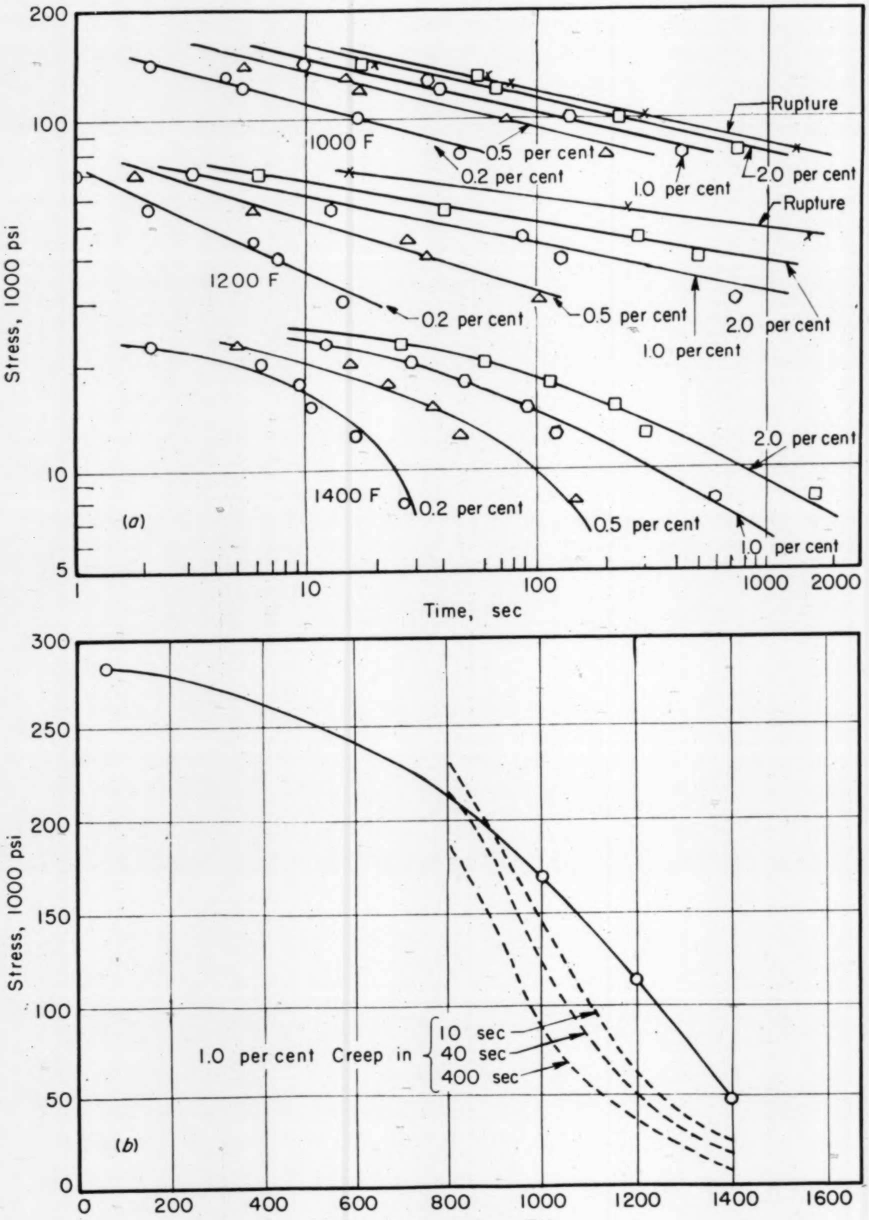
Table I shows the chemical compositions of the various alloys and the conditions of heat treatment and of cold work in which they were evaluated. All the test specimens were sheet material oriented in the longitudinal direction. With the exception of the type 304 stainless steel cold rolled at −105 F, all of the test materials are commercially available.

The type 304 cold-reduced 80 per cent at −105 F is an experimental

TABLE I.—ALLOYS EVALUATED.

Alloy	Chemical Compositions of Stainless Steels, per cent									
	C	Mn	P	S	Si	Cr	Ni	Mo	Al	Miscellaneous	Fe
Type 301[a]	0.07	1.06	0.029	0.024	0.40	17.62	7.25	0.21	...	Cu (0.23)	balance
Type 304[b]	0.08[c]	2.00[c]	0.045[c]	0.030[c]	1.00[c]	18 to 20	8 to 12	...	...	...	balance
PH 15-7 Mo[a]	0.07	0.69	0.019	0.017	0.20	15.05	7.16	2.30	1.26	...	balance
	Chemical Composition of Nickel Alloys, per cent										
	C	Mn	S	Cr	Al	Mo	Ti	Fe	Co	Cb + Ta	Ni
René[d] 41[a] nickel-chromium-molybdenum-cobalt alloy	0.13	0.05	0.02	18.9	1.74	9.96	3.03	0.17	10.78	...	balance
Inconel[e] X[b] nickel-chromium age-hardening alloy	0.04	0.50	...	14.5	0.70	...	2.5	7.0	...	1.0	balance
Nimonic[e] 90[b] nickel-chromium alloy	0.08	...	...	19.5	1.2	...	2.5	0.5	19.5	...	balance
	Condition of Alloys Evaluated										
Type 301	80 per cent cold-reduced by rolling at room temperature										
Type 304	80 per cent cold-reduced by rolling at −105 F, stress relieved 800 F 24 hr										
PH 15-7 Mo (CH 900)	cold-reduced 60 per cent at 75 F, aged 900 F 1 hr, air-cooled										
PH 15-7 Mo (RH 950)	1750 F 10 min, air-cooled, −100 F 8 hr, 950 F 1 hr, air-cooled										
René[d] 41 nickel-chromium-molybdenum-cobalt alloy	Water-quenched from 1950 F, tempered 16 hr at 1400 F, air-cooled										
Inconel[e] X nickel-chromium age-hardening alloy	50 per cent cold-reduced and aged 20 hr at 1100 F, air-cooled										
Nimonic[e] 90 nickel-chromium alloy	30 per cent cold-reduced and aged 16 hr at 1300 F, air-cooled										

[a] Heat analysis.
[b] Nominal composition.
[c] Maximum.
[d] Trade mark, General Electric Co.
[e] Trade mark, International Nickel Co., Inc.



(*a*) Time required for various amounts of creep at various stresses and temperatures.

(*b*) Effect of temperature on 1.0 per cent offset tensile yield strength at 0.1 in. per in. per sec strain rate and on stress required to reach 1.0 per cent creep strain at various times.

FIG. 1.—Type 301, 80 per cent Cold-Reduced Stainless Steel Sheet.

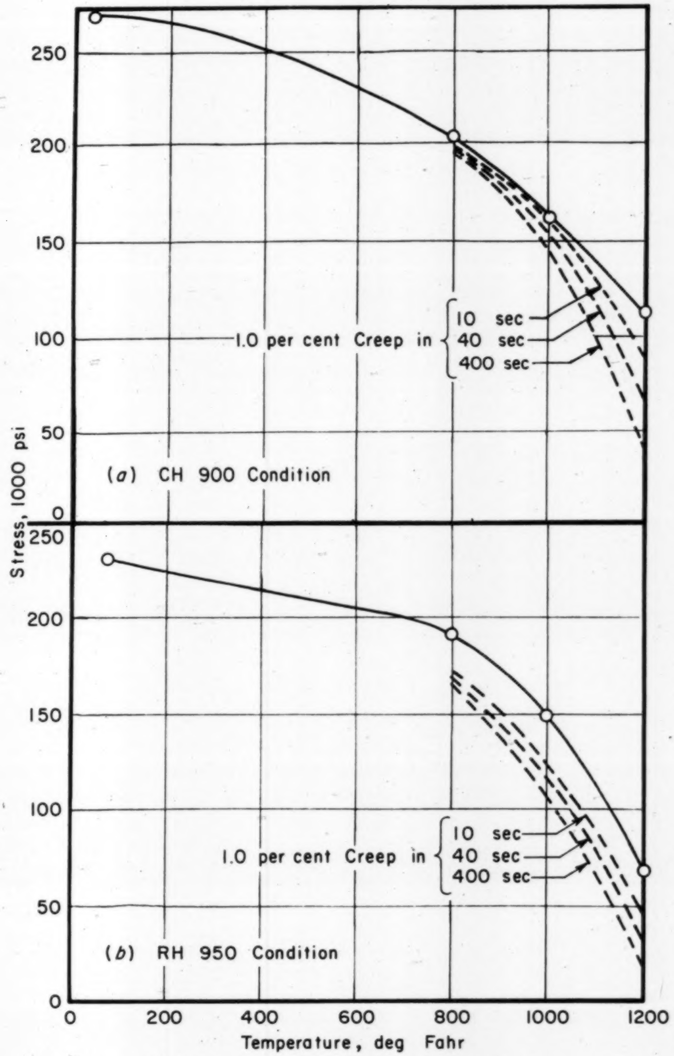


FIG. 2.—PH 15-7 Mo Stainless Steel Sheet.

Effect of temperature on 1.0 per cent offset tensile yield strength at 0.1 in. per in. per sec strain rate and on stress required to reach 1.0 per cent creep strain at various times.

material. It represents an initial attempt at Southern Research Institute to develop superior strength in austenitic stainless steels by working them at subzero temperatures. Although this phenomenon was first mentioned in the literature several years ago (**4,5**), it has never been developed into commercial application. Certain types of austenitic stainless steel are highly susceptible to work-hardening because of a certain degree of austenite-to-ferrite transformation during plastic deformation. Greater degrees of transformation, and hence greater improvement in strength, can be obtained if the deformation is carried out at subzero temperatures.

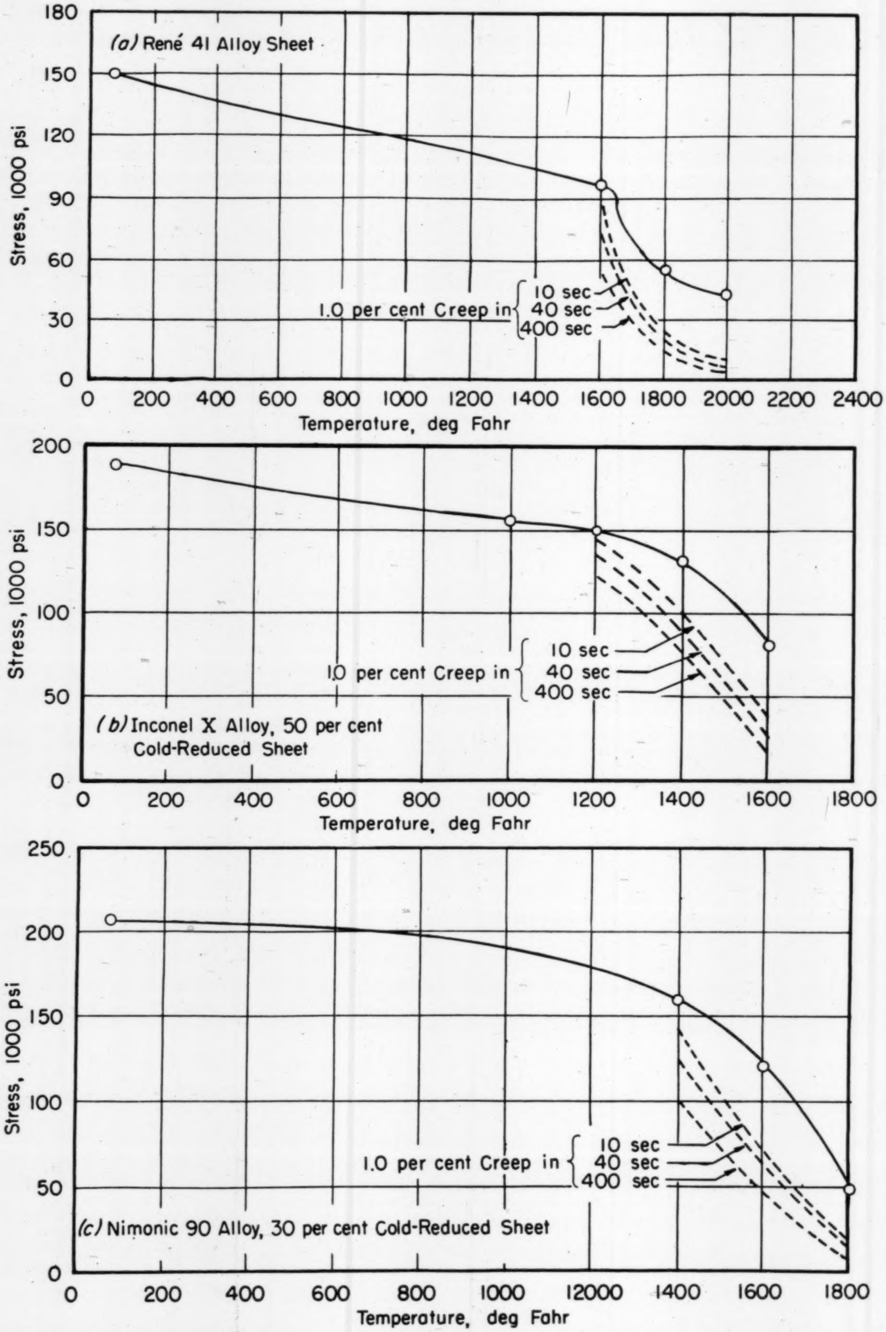


FIG. 3.—Effect of Temperature on 1.0 per cent Offset Tensile Yield Strength at 0.1 in. per in per sec Strain Rate and on Stress Required to Reach 1.0 per cent Creep Strain at Various Times.

TEST RESULTS

Figure 1(*a*) shows short-time creep data for 80 per cent cold-reduced type 301 stainless steel plotted in the form of design curves. These curves, which are representative of the type of creep data obtained for all the alloys evaluated, show the time required for 0.2, 0.5, 1, and 2 per cent creep and for rupture as functions of stress at different constant temperatures. The time scale and various degrees of creep strain pertain to time and deformation while the specimens were at constant load and constant temperature. They do not include the few seconds that elapsed or the slight amounts of elastic and plastic deformation that occurred while the specimens were being heated from room temperature to the desired constant temperatures. Similar to long-time creep strength, Fig. 1(*a*) shows that short-time creep strength increases with decreasing times under load at each temperature.

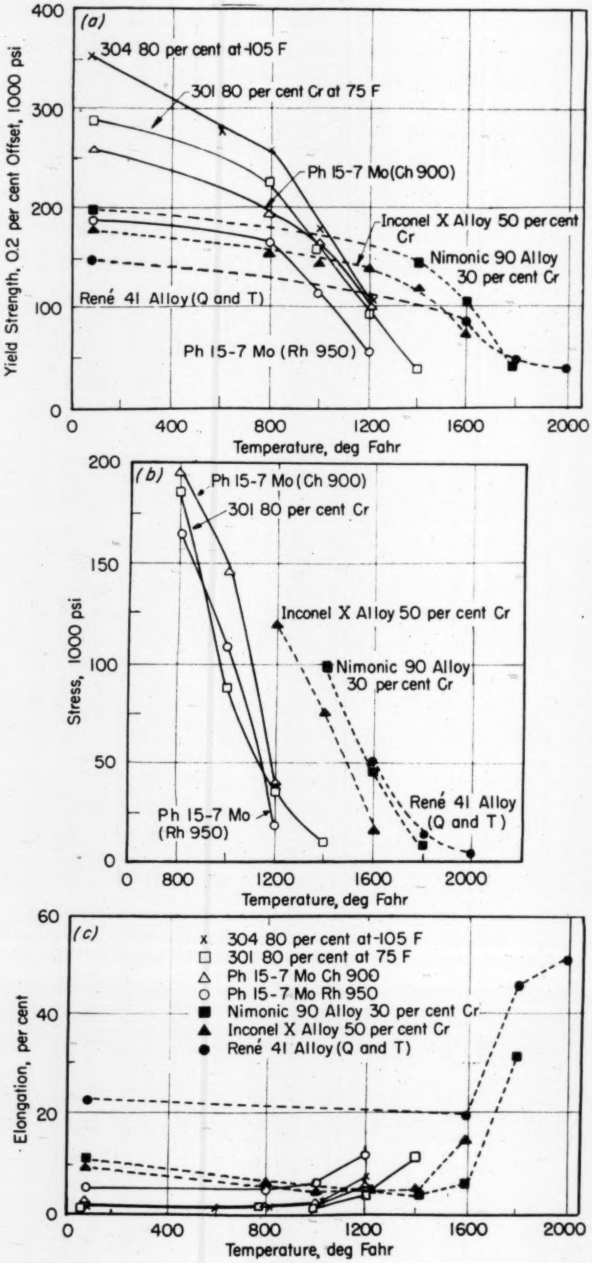
Figure 1(*b*) shows tensile and creep data combined into a single plot useful for design purposes. The solid curve represents, as a function of temperature, the 1.0 per cent offset yield strength as determined from the tensile stress-strain curves for 80 per cent cold-reduced type 301 stainless steel. The dotted curves on this plot represent the stresses required to reach 1.0 per cent creep strain at constant times of 10, 40, and 400 sec. These curves were obtained by cross-plotting stress-temperature data from Fig. 1(*a*) at various constant times and at 1 per cent creep strain. Similar plots are shown in Figs. 2 and 3 for the the other test materials with the exception of type 304 stainless, which has been tested in tension only.

The curves in Figs. 1(*b*), 2, and 3 are useful in the design of structures that must function for short periods of time with a maximum allowable permanent deformation of 1.0 per cent. The 1.0 per cent offset tensile yield strength represents the maximum allowable stress for conditions of rapid loading and unloading with no hold time at maximum stress. The dotted curves show the degree to which the allowable stresses and allowable temperatures decrease when the alloy is held at constant temperature and constant stress for various short periods of time. For the design of structures in which the design criterion is some other degree of permanent deformation or rupture, the original tensile and creep data can be used to construct plots of this type for other degrees of tensile offset and creep deformation and for ultimate tensile strength and creep rupture in times up to ½ hr.

COMPARISON

Comparisons of the 0.2 per cent offset yield strengths as functions of temperature are shown in Fig. 4(*a*) for the various alloys covered by this paper. The most striking fact brought out by this illustration is the superior strength of the type 304 stainless steel that was cold-reduced 80 per cent at −105 F, the room-temperature yield strength exceeding 350,000 psi. This strength appreciably exceeds the room-temperature yield strength of 288,000 psi in the 80 per cent cold-reduced type 301, which is one of the strongest commercially available forms of stainless steel. The strength advantage of the material rolled at −105 F is maintained as the temperature increases up to 1200 F, although the superiority decreases as the temperature is increased above 800 F. Additional investigations are being made to determine the compositions and rolling conditions for the development of optimum properties by low-temperature rolling of austenitic stainless steel.

Figure 4(*a*) also shows that the 80 per cent cold-reduced type 301 has higher yield strength than the 60 per cent cold-reduced precipitation-hardening grade, PH 15-7 Mo (CH 900), at



(*a*) On 0.2 per cent offset tensile yield strength of various sheet alloys at a strain rate of 0.1 in. per in. per sec.

(*b*) On stress required to reach 1.0 per cent creep strain in 400 sec for various sheet alloys.

(*c*) On total tensile elongation of various sheet alloys at a strain rate of 0.1 in. per in. per sec.

FIG. 4.—Effect of Temperature.

temperatures up to 1000 F; at higher temperatures, these two materials are about equal in yield strength. The yield strength of the cold-worked PH 15-7 Mo (CH 900) is higher than that of the precipitation-hardened material, PH 15-7 Mo (RH 950), throughout the entire temperature range in which they were evaluated. The stainless steels, in general, are stronger than the nickel alloys at temperatures up to about 1000 F, but the nickel alloys are stronger at higher temperatures. Of the nickel alloys, the 30 per cent cold-reduced Nimonic 90 alloy has the highest yield strength at temperatures up to about 1800 F, at which temperature its strength is slightly exceeded by that of the quenched and tempered René 41 alloy, which has a precipitation-hardened structure. The yield strength of the 50 per cent cold-reduced Inconel X alloy is intermediate to that of the Nimonic 90 alloy and René 41 alloy at temperatures up to 1500 F, at which temperature the strength of the Inconel X alloy falls below that of the René 41 alloy.

It should be remembered that all tensile strength data—1.0 per cent offset yield strength and 0.2 per cent offset yield strength—shown in this paper were obtained at a rapid strain rate of 0.1 in. per in. per sec. This rate is about 1000 times faster than the rate of 0.005 in. per in. per min normally used in tension tests. Since the strength of most metals tends to increase with increasing strain rate especially at high temperatures the tensile strength values are probably somewhat higher than those that would be obtained in ordinary tension tests.

Figure 4(*b*) shows comparisons of the short-time creep strengths of the various alloys as functions of temperature, creep strength being represented as the stress required to cause 1.0 per cent creep strain in 400 sec. This illustration shows that the nickel alloys exhibit useful short-time creep properties to considerably higher temperatures than the stainless steels do. The creep strengths of the nickel alloys in the temperature range 1400 to 1800 F are roughly equivalent to those of the stainless steels in the temperature range 1000 to 1400 F. Quenched and tempered René 41 alloy is shown to have the highest short-time creep strength of the alloys evaluated, but it is closely approached in this property by the 30 per cent cold-reduced Nimonic 90 alloy.

In contrast to the tensile yield strength comparison in Fig. 4(*a*), Fig. 4(*b*) shows that the work-hardened precipitation-hardening stainless steel, PH 15-7 Mo (CH 900), is somewhat superior in short-time creep strength to the 80 per cent cold-reduced type 301 stainless steel in the temperature range 800 to 1200 F. It is believed that the improved relative position of the PH 15-7 Mo (CH 900) is a result of its combined precipitation-hardened and work-hardened structure, whereas the type 301 was work hardened only. As in the case of the nickel alloys, work-hardening provides superior strength at low temperatures and at moderately high temperatures for short periods of time. With increasing temperatures and times, precipitation hardening tends to provide better strength. Similarly, the comparative level of the precipitation-hardened stainless steel, PH 15-7 Mo (RH 950), in Fig. 4(*b*) is considerably improved over its level in the tensile yield strength comparison, its short-time creep strength overlapping that of the cold-rolled type 301 stainless.

Figure 4(*c*) shows the total tensile elongations of the various alloys as functions of temperature. Both for the stainless steels and for the nickel alloys, the precipitation-hardened materials exhibit considerably greater tensile ductility than the work-hardened materials. At least at the lower temperatures, the inferiority in ductility of the work-hardened materials is associated with their superiority in strength. Similarly, the superiority in ductility of the nickel alloys over the stainless steels at temperatures up to about 1000 F is associated in part with lower strength levels in the nickel alloys. The nickel alloys decrease slightly in ductility with increasing temperature up to 1400 or 1600 F and then increase sharply in ductility with further increases in temperature. The stainless steels exhibit constant elongation values until the temperature is increased above 800 F, the elongation increasing with further increases in temperature.

Although the type 304 stainless that was cold rolled at −105 F has considerably higher tensile strength than the stainless steels that were cold worked at room temperature, Fig. 4(*c*) shows that its elongation is equal to or higher than those of the 80 per cent cold-reduced type 301 and the PH 15-7 Mo (CH 900).

CONCLUSIONS

1. The load-carrying capacity of structural sheet metals at elevated temperatures increases as the time of exposure to load decreases from 30 min to 1 sec.

2. High-strength stainless steels, in general, have higher tensile strengths than nickel alloys at temperatures up to about 1000 F. At higher temperatures, the nickel alloys have higher tensile strength.

3. The nickel alloys exhibit useful short-time creep properties to considerably higher temperatures than the stainless steels do, the short-time creep strengths of the nickel alloys in the range 1400 to 1800 F being roughly equivalent to those of the stainless steels in the range 1000 to 1400 F.

4. The results of preliminary experiments indicate that cold rolling of certain austenitic stainless steels at subzero temperatures can produce markedly higher short-time strength than equivalent rolling reductions carried out at room temperature.

5. Both for stainless steels and for nickel alloys, cold-worked structures are superior to precipitation-hardened structures in strength at low temperatures and for short rupture times at moderately elevated temperatures. With increasing temperatures and times under load the strengths of the cold-worked structures are approached or exceeded by those of the precipitation-hardened structures.

Acknowledgments:

The work was carried out at Southern Research Institute under the sponsorship of and in cooperation with the International Nickel Co., Inc. Many members of the Metallurgy Division at Southern Research Institute assisted in the work, J. B. Preston, J. D. Morrison, D. E. Rice, and W. L. Rogers being the main contributors.

REFERENCES

(1) James B. Preston, William P. Roe, and J. Robert Kattus, "Determination of the Mechanical Properties of Aircraft-Structural Materials at Very High Temperatures after Rapid Heating," Wright Air Development Center, Technical Report 57-649, Part I, Nov. 1957.

(2) J. R. Kattus, "Structural Materials for

Missile Applications at Very High Temperatures," *Jet Propulsion*, June 1957, pp. 644–649.

(3) J. R. Kattus, "Effect of Holding Time and Strain Rate on the Tensile Properties of Structural Metals," Short-Time High-Temperature Testing, Am. Soc. Metals, 1958.

(4) V. N. Krivobok and A. M. Talbot, "Effect of Temperature on the Mechanical Properties, Characteristics, and Processing of Austenitic Stainless Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 895 (1950).

(5) N. A. Zeigler and P. H. Brace, "Hardening of Austenitic Stainless Steels by Mechanical Working at Subzero Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 861 (1950).

COMPRESSION TESTING OF SHEET MAGNESIUM UTILIZING RAPID HEATING*

By Raymond W. Fenn, Jr.[1]

Synopsis

This paper deals with the design, construction, and performance of test equipment for determining compressive stress - strain curves and short-time elevated-temperature compression-creep properties of sheet metals under conditions of rapid heating. The following are some of the problems encountered in this work: supporting the test specimen, minimizing frictional loads, aligning the stress and specimen axes, heating the test specimens rapidly by the resistance-heating method and controlling thermal gradients. Techniques developed and believed satisfactory for dealing with these problems are described as well as certain techniques which were unsatisfactory.

A comparison between the compression and tension short-time creep strengths of HK31A-H24 magnesium alloy sheet shows that the tensile creep strength is somewhat higher than the compressive creep strength at certain elevated temperatures. The data also show that HK31A-H24 produced by a process designated in the text as process "B" has higher short-time elevated-temperature properties than HK31A-H24 produced by a process designated as process "A."

The increased use of magnesium alloys in the monocoque construction of missiles has created a demand for the elevated-temperature compressive properties of these alloys obtained under rapid-heating conditions. Although considerable elevated-temperature work, utilizing relatively slow-heating rates, has been done on the determination of compressive yield strength (**1–12**)[2] and to a lesser extent on compressive creep strength (**13–17**) the author is aware of only one reference (Bernett (**18**)) to the use of rapid-heating techniques for testing sheet compression specimens and this reference gives very little information on the problems encountered or the solution to these problems. The fluid-heating method of McGill et al (**19**) could presumably be adapted for compression testing magnesium sheet alloys. However, this method appears to be limited to test temperatures on the order of 600 F because of the relatively low decomposition temperatures of oils and the difficulty of obtaining a safe noncorrosive salt bath for use at higher temperatures with magnesium alloys. The equipment described in this paper was developed specifically for the determination of compressive isochronous-stress-strain curves of magnesium sheet alloys at temperatures of 300 to

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Chief, Testing and Instrumentation, Metallurgical Laboratory, The Dow Metal Products Co., Division of The Dow Chemical Co., Midland, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

1000 F for times of 5 sec to 15 min and strains up to 2 per cent subsequent to rapid heating by the resistance-heating method. The rapid-heating technique, although developed for use with creep equipment, can be used with a conventional testing machine to obtain compression yield strength and compression stress-strain curves.

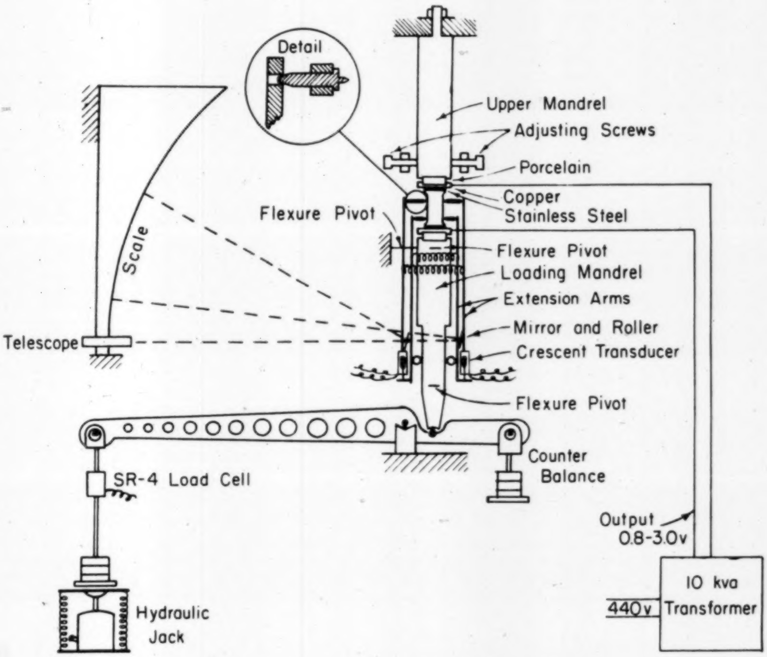
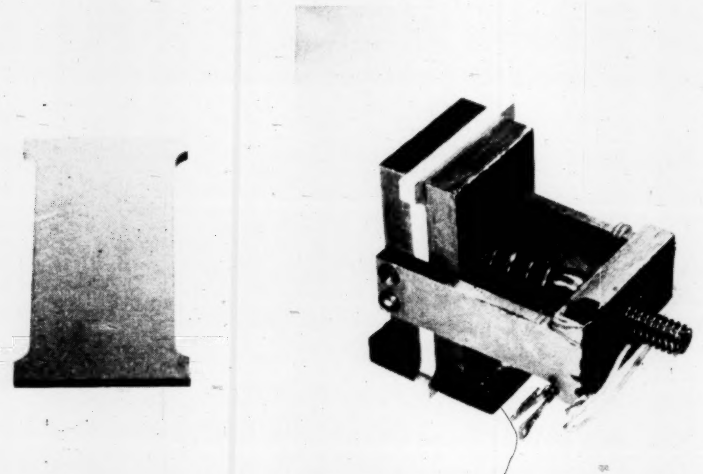

Detail
Upper Mandrel
Adjusting Screws
Porcelain
Copper
Stainless Steel
Flexure Pivot
Flexure Pivot
Loading Mandrel
Extension Arms
Mirror and Roller
Crescent Transducer
Flexure Pivot
Scale
Telescope
SR-4 Load Cell
Counter Balance
Output 0.8-3.0v
10 kva Transformer
440v
Hydraulic Jack

FIG. 1.—Rapid-Heating Compression - Creep Equipment.

The measurement of short-time tensile creep properties using high-speed resistance heating is a relatively simple problem compared to the measurement of compressive creep properties under the same test conditions. This statement is not intended to minimize the problems of tension testing with high-speed heating equipment but rather to point out that the same problems occur in compression testing but are in general more severe. For example, misalignment of stress and specimen axes to an extent which might be tolerable in tension is accentuated by increasing stress and may cause buckling in compression testing. The common practice of lengthening the tension test specimens to obtain smaller temperature gradients cannot be used to the same extent in compression testing. Other problems of testing compression specimens with the resistance-heating technique are: minimizing frictional loads resulting from supporting the specimen, minimizing the effects of thickness changes in the specimen resulting from thermal expansion and the Poisson effect, transferring the electrical current from the bus bars to the specimen with minimum contact resistance and making heat losses from the specimen as uniform as possible to obtain satisfactory temperature gradients and control.

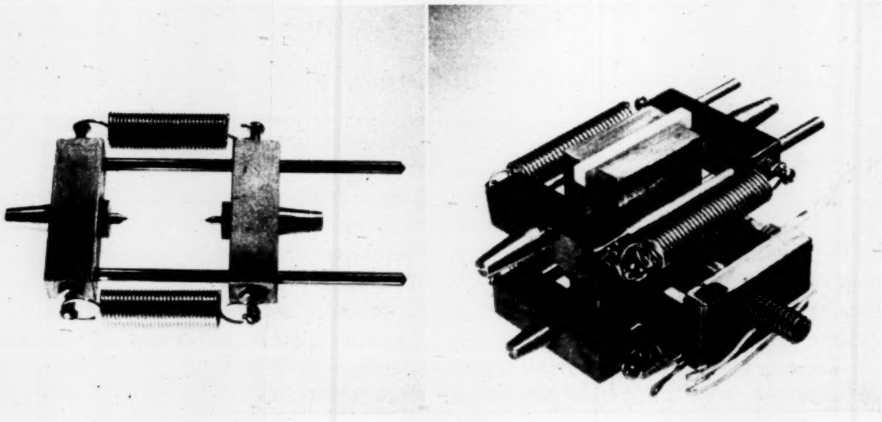
DESCRIPTION OF EQUIPMENT

The testing machine, a schematic sketch of which is given in Fig. 1, consists of a tension-creep machine which was rebuilt to permit direct application of compressive loads by using a lever with a 20:1 mechanical advantage. The upper loading mandrel is bolted to the top of the creep frame and positioned at the lower end by four screws extending from a supporting frame. This arrangement increases the rigidity of the upper mandrel and provides a method of adjusting the alignment of the compressive faces of the upper and lower mandrels. The motion of the lower mandrel is guided by flexure pivots and the lever knife edge.



(*a*) Test specimen $1\frac{1}{4}$ by 2 in. with a reduced section 1 by $1\frac{1}{2}$ in.

(*b*) Stainless steel specimen-supporting jig showing compression spring.



(*c*) Stainless steel compressometer collar.

(*d*) Specimen in supporting jig with compressometer collars attached.

FIG. 2.—Details of Specimen, Specimen-Supporting Jig and Compressometer Collars.

SR-4 load cells of various capacities are placed in the bale pan-lever linkage to serve as load measuring transducers. A hydraulic jack or electric screw jack is used to transfer the entire load on the bale pan from the jack to the test specimen in a continuous, rather than stepwise, operation.

SPECIMEN SUPPORT

An important variable in any compression test is the method of supporting the test specimen as demonstrated by the room temperature tests of Kotanchik (**20**). Minimizing frictional loads from the supports (needed to prevent buckling) and obtaining uniform dissipation of heat from the test specimen are major problems in compression tests using rapid heating. In an attempt to satisfy both of these conditions, a lubricated flat-plate type of support was selected as more desirable than rollers, balls, leaf springs or grooved plates described in the literature (**8,12, 20,21**).

The test specimen (Fig. 2(*a*)) is laterally supported during testing with a stainless steel jig (Fig. 2(*b*)) consisting of two supporting plates, a clamping yoke, a steel compression spring and a clamping screw. The supporting plates are surface-ground and polished on a No. 0000 metallographic paper to provide smooth flat surfaces for contact with the specimen. The centrally located clamping yoke and single clamping-pressure adjustment screw permit uniform clamping pressure to be obtained and facilitate the measurement of the clamping load. The clamping load at both room and elevated temperatures was measured by attaching SR-4 strain gages on the clamping yoke adjacent to the adjusting screw. The use of a spring with the adjusting screw not only permits more accurate adjustment of the clamping load but minimizes the changes in clamping load and therefore changes in frictional loads and restraints caused by increases in specimen thickness resulting from thermal expansion and the Poisson effect. Measurements of jig clamping force show that the clamping force remains constant upon heating the specimen to 500 F. However, there is a gradual increase in clamping force of approximately 1 lb upon holding at 500 F for 5 min, apparently resulting from thermal expansion of the spring and adjusting screw. Compressing the specimen 2 per cent also increases the clamping force approximately 1 lb. These changes in clamping force are considered to have a negligible effect on the test results, based on the data discussed in the following paragraph.

An indication of the relative frictional loads with various clamping loads was obtained by measuring the maximum load (termed static frictional load) required to move a specimen vertically in the supporting jig. Although this load is probably not an exact measurement of the load error resulting from supporting the specimen during a test, it appears to be a better criterion for evaluating the method of specimen support and effect of lubrication than measurements of Young's modulus and compressive yield strength since these properties can be significantly affected by other test variables. The frictional loads observed with and without graphite lubrication at temperatures of 75, 500, and 800 F are shown in Fig. 3 as a function of support-jig clamping force. These data indicate: (1) that the frictional loads are significantly lower and less affected by temperature when dry graphite powder is used as a lubricant and (2) that a surface ground finish (approximately 32 microinches) on the support jig gives essentially the same results as a polish on No. 0000 metallographic paper. In tests on HK31A-H24 performed at a stress level of 21,000 psi, a clamping force of 40 lb results in load errors on the order of 0.4 and 1.4 per cent for a 0.125-in. thick specimen and 0.8 and 2.8 per cent for 0.062-in. thick specimens with and without graphite lubricant respectively.

The static frictional data in Fig. 3 were obtained using: (1) support plates made of type 304 stainless steel, (2) $\frac{1}{8}$-in. thick HK31A-H24 sheet specimens with surfaces in the "as-received" condition (contact area = 4 sq in.) and (3) the compressive creep equipment. Dynamic frictional values obtained at approximately 0.010 in. per min with a testing machine were on the order of 20 per cent lower than the static frictional values.

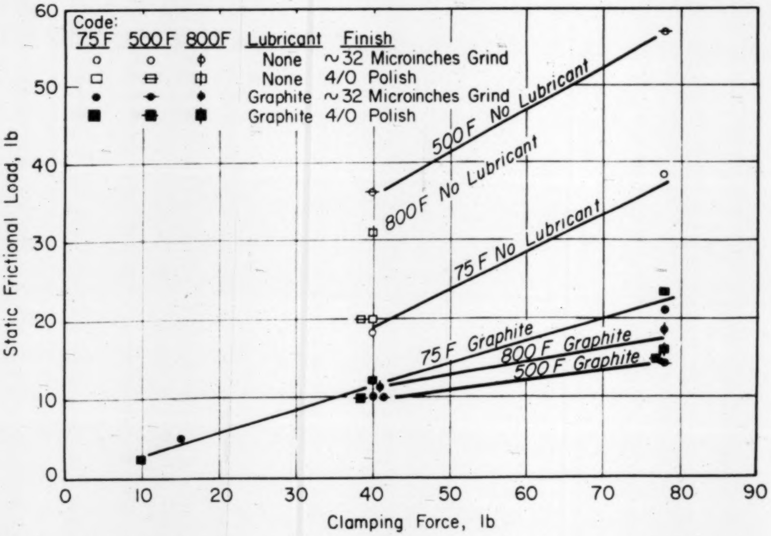


FIG. 3.—Effect of Clamping Force on Static Frictional Load at 75, 500, and 800 F with and Without Graphite Lubricant.

A clamping force of 40 lb is sufficient to prevent buckling $\frac{1}{8}$-in. HK31A-H24 sheet tested with or without graphite lubricant at 400, 500, and 600 F and stress levels as shown in Table I using an unsupported specimen length of 0.060 in. Testing at higher stress levels, which would be encountered at lower temperatures, would undoubtedly require a clamping force higher than 40 lb.

ALIGNMENT OF STRESS AND SPECIMEN AXES

Good alignment between the stress and specimen axes and between the ends of the specimen and the faces of the compressive mandrels is essential for accurate compression test results, particularly Young's modulus. The use of a subpress to obtain accurate alignment was considered and rejected because of the possibility of frictional problems from nonuniform heating which might accompany the rapid heating of the test specimen. Instead of using a subpress, a means of aligning the upper mandrel face with that of the lower mandrel was designed into the equipment and a means of centering the specimen on the lower mandrel was provided.

The supporting jig containing the test specimen is accurately centered on the lower compression mandrel by using a copper locating plate (Fig. 4(*f*)) which also serves as an electrical bus bar. The locating plate is held in place by a 1.500-in. diameter Lapp chemical por-

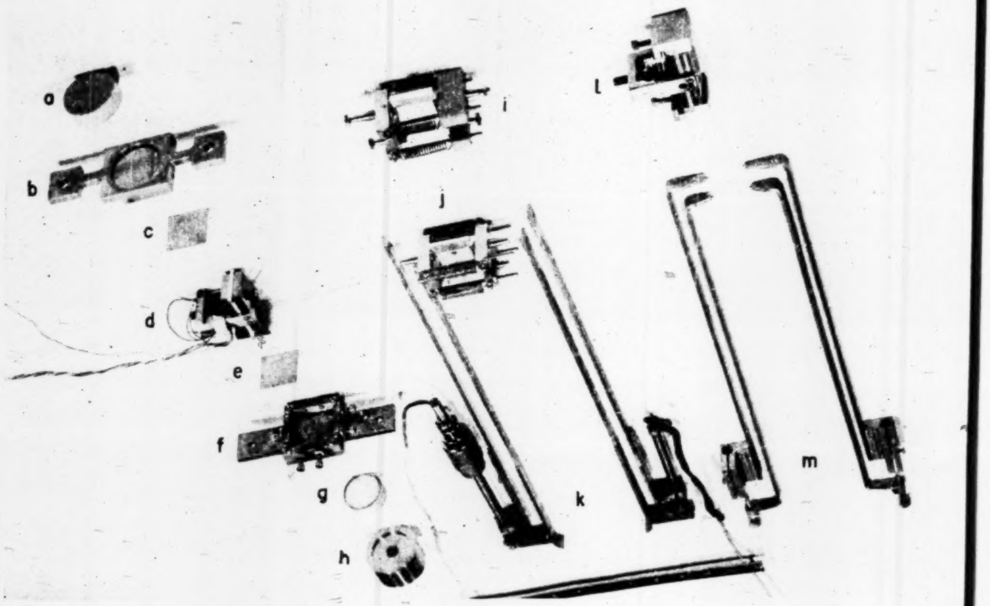
TABLE I.—SHORT-TIME COMPRESSION CREEP DATA FOR HK31A-H24^a (LOT 51454)

TABLE I.—SHORT-TIME COMPRESSION CREEP DATA FOR HK31A-H24[a] (LOT 51454).

Specimen	Test Temperature, deg Fahr	Stress, psi	Time at Temperature, sec	Strain Rate, in. per in. per min	Heating Rate, deg Fahr per sec	Loading Time, sec	Loading Strain, per cent	Per Cent Total Strain at Creep Times of:									Creep Time, sec, for Per Cent Creep Strain of:					
								Sec				Min										
								5	10	15	30	1	2	5	10	15	0.1	0.2	0.3	0.4	0.5	1.0
No. 1	400	12 000	56	0.0345	27.1	4	0.230	0.230	0.235	0.240	0.240	0.240	0.240	0.240	0.250	0.260	...	...	...	...	...	...
No. 2	400	14 000	70	0.0405	25.0	4	0.270	0.290	0.290	0.295	0.295	0.295	0.300	0.305	0.305	0.305	...	...	...	...	...	...
No. 3	400	16 000	78	0.0335	27.1	5	0.280	0.285	0.300	0.300	0.300	0.300	0.300	0.300	0.305	0.305	...	...	...	...	...	...
No. 4	400	18 000	64	0.0310	23.2	6	0.310	0.320	0.320	0.320	0.320	0.320	0.330	0.340	0.360	0.370	...	...	...	...	...	...
No. 5	400	20 000	35	0.0342	21.7	6.5	0.370	0.390	0.390	0.395	0.395	0.395	0.400	0.420	0.440	0.460	...	...	...	...	...	...
No. 6	400	22 000	69	0.0315	25.0	8	0.420	0.445	0.450	0.450	0.455	0.460	0.470	0.495	0.510	0.520	900	...	...	...	...	...
No. 7	400	24 000	68	0.0346	25.0	9	0.520	0.570	0.590	0.600	0.610	0.620	0.640	0.680	0.720	0.745	66	576	...	...	...	...
No. 8	400	25 000	90	0.0378	21.7	9	0.560	0.640	0.655	0.665	0.680	0.700	0.725	0.775	0.810	0.835	12	226	900	...	...	...
No. 9	400	26 000	618	0.0456	23.2	9	0.760	0.940	0.970	0.985	1.020	1.050	1.085	1.130	1.175	1.200	1	9	66	464	...	...
No. 10	400	27 000	583	0.0550	20.4	10	0.970	>2.0	...	...	...	...	...	...	...	...	...	...	...	...	...	...
No. 11	500	8 000	61	0.0240	10.1	3	0.120	0.130	0.130	0.130	0.140	0.145	0.155	0.170	0.190	0.210	1140	...	...	...	...	...
No. 12	500	12 000	120	0.0322	19.3	4	0.215	0.220	0.220	0.220	0.235	0.240	0.255	0.280	0.300	0.320	836	...	...	...	...	...
No. 13	500	14 000	55	0.0300	21.0	5	0.250	0.260	0.263	0.263	0.280	0.295	0.310	0.343	0.390	0.430	310	1000	...	...	...	...
No. 14	500	16 000	100	0.0267	8.4	7	0.310	0.340	0.350	0.360	0.385	0.415	0.465	0.560	...	...	52	186	446	...	...	...
No. 15	500	18 000	125	0.0285	9.3	8	0.380	0.410	0.420	0.440	0.460	0.510	0.570	0.670	0.805	0.910	40	130	306	530	790	...
No. 16	500	20 000	127	0.0338	11.3	8	0.450	0.510	0.540	0.560	0.610	0.710	0.850	1.080	1.350	1.600	13	42	72	116	193	732
No. 17	500	21 000	48	0.1500	9.3	2	0.500	0.620	0.640	0.660	0.710	0.820	0.950	1.200	1.460	1.690	2	28	52	92	146	410
No. 18	500	21 000	285	0.0480	7.0	6	0.480	0.560	0.590	0.620	0.670	0.780	0.910	1.160	1.450	1.710	8	32	60	105	160	226
No. 19	500	21 000	50	0.0450	7.0	6	0.450	0.580	0.610	0.640	0.700	0.800	0.930	1.180	1.470	1.750	2	18	42	80	133	576
No. 20	500	21 000	180	0.0290	9.3	10	0.480	0.560	0.590	0.620	0.710	0.850	1.040	1.400	1.910	>2.0	8	25	43	65	93	344
No. 21	500	22 000	75	0.0408	9.3	10	0.680	0.770	0.820	0.850	0.920	1.050	1.250	1.570	1.900	>2.0	5	22	43	67	95	385
No. 22	500	23 000	79	0.0425	9.3	11	0.780	0.900	0.970	1.030	1.160	1.360	1.620	>2.0	...	...	3	10	20	32	48	160
No. 23	500	24 000	30	0.0480	11.4	12	0.960	1.140	1.180	1.240	1.400	1.790	>2.0	...	...	...	1	8	16	26	35	68
No. 24	600	3 000	75	0.0160	10.6	1.5	0.040	0.055	0.060	0.060	0.065	0.070	0.100	0.145	0.200	0.270	274	566	...	...	...	...
No. 25	600	4 000	74	0.0300	13.0	1.0	0.050	0.080	0.085	0.085	0.105	0.140	0.205	0.360	0.620	0.885	65	175	288	406	526	1020
No. 26	600	5 000	120	0.0300	8.7	2.0	0.100	0.115	0.130	0.140	0.170	0.220	0.300	0.510	0.850	1.200	46	120	204	294	381	814
No. 27	600	6 000	83	0.0300	12.5	2.0	0.100	0.130	0.135	0.145	0.185	0.250	0.355	0.660	1.160	1.680	35	88	143	204	262	555
No. 28	600	7 000	77	0.0361	13.7	2.0	0.120	0.170	0.190	0.205	0.250	0.340	0.495	0.990	1.890	>2.0	20	56	92	128	159	352
No. 29	600	8 000	80	0.0223	13.5	3.5	0.130	0.160	0.180	0.205	0.265	0.375	0.560	1.070	2.000	>2.0	21	46	77	110	145	319
No. 30	600	9 000	145	0.0270	5.3	4.0	0.180	0.230	0.265	0.300	0.400	0.575	0.880	1.775	>2.0	...	11	27	42	61	80	184
No. 31	600	10 000	71	0.0400	13.1	3.0	0.200	0.280	0.340	0.390	0.535	0.825	1.350	>2.0	...	...	6	16	25	36	46	102
No. 32	600	11 000	125	0.0382	9.6	4.0	0.255	0.340	0.400	0.465	0.675	1.030	1.645	>2.0	...	...	7	15	22	29	37	80
No. 33	600	12 000	30	0.0342	7.4	5.0	0.285	0.395	0.455	0.520	0.700	1.080	1.980	>2.0	...	...	4	12	20	28	36	74

[a] Produced by process "B."

celain disk (Fig. 4(*g*)) which is ground to tolerances of 0.0005 in. to fit 0.1-in. deep recesses in the lower mandrel and the locating plate. This porcelain disk serves as both an electrical and thermal insulator in the system.



(*a*) Porcelain insulator.
(*b*) Upper mandrel bus bar plate.
(*c*) Stainless steel resistor.
(*d*) Specimen supporting jig showing thermocouples clamped in place.
(*e*) Stainless steel resistor.
(*f*) Copper locating plate.
(*g*) Porcelain insulator.
(*h*) Lower mandrel adapter.
(*i*) Temperature-measuring compressometer collars.
(*j*) Stainless steel compressometer collars.
(*k*) Stainless steel extension arms with Crescent Engineering transducers.
(*l*) Stainless steel resistor vises mounted on specimen with supporting jig.
(*m*) Stainless steel extension arms.

FIG. 4.—Compression - Creep Testing Accessories.

The specimen is centered on the locating plate by rotating the locating plate on the porcelain disk while eccentricity measurements are being made on the specimen and jig at 180 deg intervals with a dial gage. The screws in the locating plate are adjusted to bring the center of the specimen within approximately 0.002 in. of the center of the lower mandrel.

The alignment of the compression faces is checked with Johansson gage blocks, and any adjustment necessary to obtain parallelism is made with the adjusting screws at the bottom of the upper mandrel. If a room temperature trial run shows elastic strain readings on opposite sides of the specimen to be more than 15 per cent different from the average reading, further adjustments are made prior to heating the specimen.

STRAIN AND LOAD MEASUREMENT

Two methods of measuring strain are provided: (1) a modified Marten's optical lever system, and (2) a Crescent Engineering measuring system. The Marten's system provides: (*a*) a means of checking the axial alignment of the stress and specimen axes (in one plane) since separate strain readings can be taken on opposite sides of the specimen, (*b*) high accuracy strain measurements for the accurate determination of Young's modulus, (*c*) a means of checking the strain measurements of the Crescent Engineering measuring system during the test and (*d*) a convenient secondary calibration standard. The Crescent Engineering linear variable permeance transducers are used for making all strain measurements which are to be autographically recorded. In both systems the strain measurements are taken over a 1-in. gage length with the same set of compressometer collars (Fig. 4(*j*)) and extension arms (Fig. 4(*k*)), simultaneously or separately as desired. The strain transfer system using: (1) the extensometer collars shown in Fig. 4(*j*), (2) tapered pin - nontapered hole connections between collars and extension arms (see detail sketch in Fig. 1) and (3) a combination of springs and rollers to support the extension arms was shown in previous work (**22**) to transfer strain with an accuracy of ±1 microinch per in. The Marten's type measuring system is essentially the same as that previously described (**22**) except that two scales are provided to give strain measuring accuracies of 4 and 10 microinches per in. The Crescent Engineering strain measuring system, SR-4 load measuring system, and accompanying instrumentation duplicate that described by Clapper (**23**).

Two types of extension arms, shown in Fig. 4(*k*) and Fig. 4(*m*) were made in both aluminum and type 304 stainless steel and tested at 500 F. The aluminum extension arms are unsatisfactory because of high thermal conductivity and high coefficient of thermal expansion which prevents the measuring system from attaining thermal equilibrium in the short time (approximately 1 min) necessary. However, the use of stainless steel in the arms does provide a strain measuring system which is stable at 500 F. The stainless steel extension arms shown in Fig. 4(*k*) show a thermal drift of less than 25 microinches per in. after a holding time of 1 min at 500 F, while the stainless steel arms in Fig. 4(*m*) show drift on the order of 175 microinches per in. under the same conditions. The improved performance of the arms shown in Fig. 4(*k*) over those shown in Fig. 4(*m*) is attributed to the low thermal conductivity of the tapered pin - nontapered hole connection between the collars and arms.

HEATING AND TEMPERATURE CONTROL

One of the major objectives in the development of this equipment was to combine rapid heating (heating times on the order of 1 min or less) with sheet compression testing to provide compression design data under conditions of interest to missile designers. The method selected to heat the specimens was the resistance-heating method in which the specimens are heated by passing high-amperage alternating current longitudinally through them. A 10-kva Westinghouse Furnatron control system, previously described by Clapper (**23**) and Fenn (**24**), permits the temperature of the specimen at the control point to be held within ±2 F.

The establishment of minimum temperature gradients within the gage length of the specimen is the principal problem to be overcome when rapid heating methods are used. In this case the compression mandrels and copper plates conducting electricity to the specimen are major heat sinks which must either be insulated from the system or provided with a separate source of heat to minimize the heat drain from the specimen. It was decided: (1) to make the mandrels out of stainless steel in order to minimize thermal conductivity, (2) to insulate the mandrels from the copper plates, and (3) to supply additional heat at the ends of the specimen by using stainless steel between the copper plates and the specimen to act as an electrical resistor. Lapp chemical porcelain is used as an insulator to minimize heat losses from the copper plates into the stainless steel mandrels. Pyrex glass and reinforced plastic are both unsatisfactory in this application.

A series of stainless steel resistors $1\frac{1}{4}$ in. square and varying in thicknesses from 0.050 to 0.250 in. were made to compensate for the heat losses at the ends of the specimens. Selection of the proper resistor for use with the various alloys, sheet thicknesses, and test temperatures is a matter of experience. However, for illustration, the use of resistors 0.100-in. thick satisfactorily compensates for the heat losses at the ends of a 0.125-in. thick specimen of HK31A-H24 alloy tested at 500 F and gives a maximum temperature difference of 10 F over the gage length after heating for 1 min.

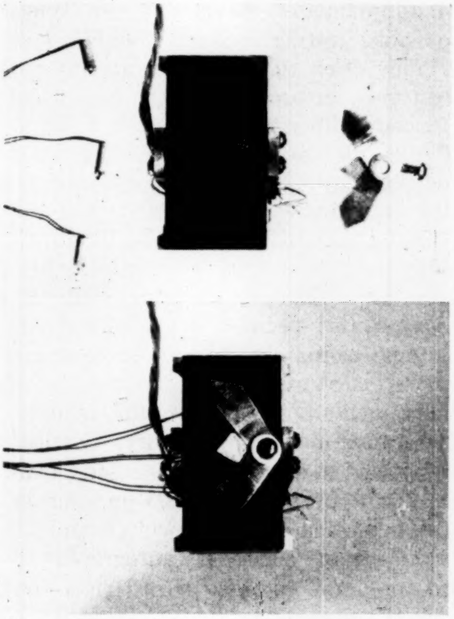
One of the most troublesome problems encountered was that of minimizing contact resistance between the test specimen, stainless steel resistors, and the copper plates supplying power. Poor mating surfaces or excessively oxidized surfaces lead to hot spots which can prevent the attainment of satisfactory temperature control. The problem of high contact resistance can be minimized by: (1) using the alignment and adjustment procedures described above for alleviating the problem of poor mating surfaces, (2) silver plating the contact surfaces, and (3) polishing the contact surfaces with No. 0 metallographic paper between tests.

Another method used to lower contact resistance between the specimen and the stainless steel resistors is to increase the contact area by making the resistors in the form of vises (Fig. 4(*l*)) and clamping them to the ends of longer specimens. Although these vises completely eliminate arcing between the specimen and resistor, it is more difficult to obtain satisfactory alignment of the stress and specimen axes and to prevent buckling when the vises shown in Fig. 4(*l*) are used instead of the resistor plates (Fig. 4(*c*, *e*)).

Contact resistance is also affected by the applied stress on the system. Although a small stress of 40 to 800 psi is applied to the specimen prior to and during heating, a drop in the temperature of the test specimen is usually noted when the specimen is completely loaded. This is attributed to a decrease in contact resistance. In tests where the contact resistance is properly controlled the drop in temperature is approximately 5 F or less which is considered acceptable. In general the recovery time is equal to or less than the time required to heat the specimen.

The stainless steel specimen-supporting jig was considered as a heat sink which might restrict the maximum heating rates attainable. Therefore, 0.010 in. resin-bonded mica electrical insulation was placed between the specimen and supporting jig as both a thermal and electrical insulator. The use of this insulating material was soon discarded when questions arose concerning the effect of its nonuniform thickness on the uniformity of clamping pressure, the effect of the resinous binder on the friction between the specimen and supporting jig and the role of this relatively soft material in some premature buckling failures which were encountered. No detrimental effects were noted on either the longitudinal or transverse temperature gradients or the rate of heating when the mica insulation was omitted. Temperature measurements during testing without insulation showed the outer surfaces of the jig to be within approximately 10 F of the 500 F test temperature after heating to the test temperature in approximately 1 min and within approximately 3 F after holding the specimen at 500 F for 2 min.

(*a*) Glass covered No. 30 I-C thermocouples with ceramic insulators ready for installation.



(*b*) Thermocouples clamped in position with leaf springs.

FIG. 5.—Technique for Clamping Thermocouples.

TEMPERATURE MEASUREMENT

Iron-constantan thermocouples were used with Brown expanded scale and multipoint recorders to measure thermal gradients in the test specimen and to monitor the temperatures. Three methods were used for attaching the thermocouples to the specimen: (1) capacitance welding (see Fig. 4(*j*)), (2) clamping in place with leaf springs (see Fig. 5(*b*)), and (3) clamping iron and constantan probes onto the edge of the specimen as the gage points (see Fig. 4(*i*)). Longitudinal and transverse thermal gradients were measured with 30-gage iron-constantan thermocouples capacitance welded centrally on the face and edge of the specimen at the midpoint and ends of the gage length to determine the maximum temperature difference within the gage length. Comparison of the edge temperatures to those in the center of the face of the specimen showed the former to average 5 F lower than the latter. Comparison of the temperature readings along the gage length of the specimen indicates that possible variations in contact resistance can cause significant temperature variations within the gage length and makes it absolutely necessary to monitor the temperature at the center and ends of the gage length on every test. Under proper operating conditions temperature gradients in the gage length can be held to 10 F or less. However, variations of ±10 F are considered acceptable for rapid heating whereas variations of ±2 F would be expected for slow heating such as is obtained with a furnace.

Comparison of the temperatures obtained with 30-gage iron-constantan thermocouples clamped against the specimen to those welded to the specimen showed no difference. The clamped-on thermocouples (Fig. 5) are now used in data gathering tests to avoid the difficulty of having to weld thermocouples to the specimen through small holes $\frac{1}{4}$-in. deep in the side of the supporting jig.

Comparison of temperatures obtained under operating conditions with the 30-gage iron-constantan thermocouple welded to the specimen and the temperatures obtained with the iron and constantan points in the compressometer collars (Fig. 4(*i*)) showed the latter to be approximately 25 F lower than the former in 500 F tests since the measuring collars are colder than the test specimen. This indicates that these temperature-measuring compressometer collars should be calibrated as a function of temperature and exposure time to be useful—something which has not been done. The temperature measuring compressometer collars (Fig. 4(*i*)) are made essentially the same as the standard stainless steel ones (Fig. 4(*j*)) except that the points for attaching the collars to the specimen are made of iron and constantan in lieu of stainless steel and two of the supporting blocks (on the right) are made of reinforced silicone plastic and used in conjunction with glass rollers between the extension arms to prevent electrically shorting the thermocouples.

PROCEDURE

The faces of the compression specimen and the supporting jig plates are rubbed in dry graphite lubricant. The specimen is placed in the supporting jig and the desired clamping pressure (usually 40 lb) is applied through a compression spring and checked by means of SR-4 strain gages on the clamping yoke. Centering the specimen with respect to the axis of the lower mandrel is accomplished by adjusting screws in the locating plate. The compressometer collars are then placed on the specimen so that the gage points rest in the punch marks made with a gage marker. The locating plate, with the supporting jig and specimen, is placed on the lower mandrel and the strain measuring system assembled. Should elastic loading at room temperature reveal significant differences in strain readings on opposite sides of the specimen, necessary adjustments are made on the upper mandrel. A stress of 800 psi or less is applied to the specimen to hold it in position and the adjusting screws in the locating plate are backed off. Then the specimen is heated in approximately 30 sec to 1 min. When possible, testing is started within 1 to 2 min after the specimen attains the test temperature. (Specimens Nos. 9 and 10 were deliberately held at 500 F for 10 min prior to testing for calibration purposes.) As experience is gained with the equipment it is expected that this holding time will be minimized and possibly held to 1 min. If relatively high accuracy Young's modulus data are desired, the specimen is loaded stepwise in approximately 1 min or less and strain measured to an accuracy of 4 microinches per in. If a routine modulus determination is satisfactory, the load is applied in approximately 5 sec (strain rate = approximately 0.03 in. per in. per min) with a hydraulic jack and the stress-strain curve is autographically recorded on an X-Y recorder. A continuous strain-time record is obtained autographically from the start of heating to the end of the test to permit the determination of short-time creep data.

RESULTS AND DISCUSSION

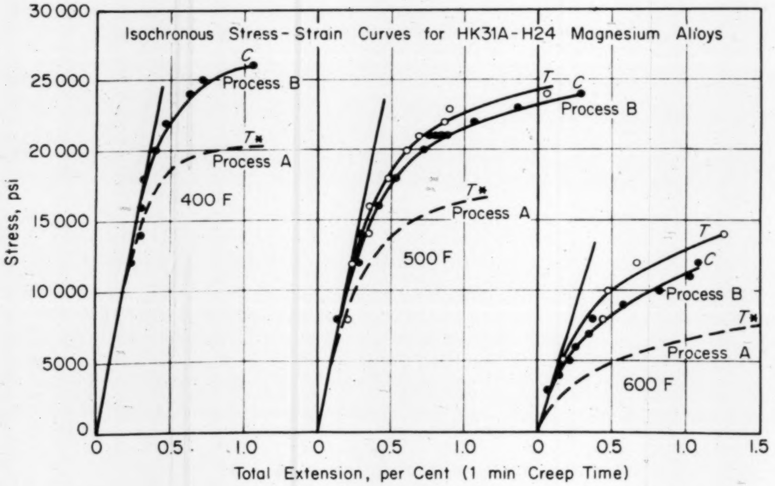
Short-time compression creep test results obtained on 0.125-in. HK31A-H24 sheet at 400, 500, and 600 F are presented in Table I. Comparison among the test results obtained for specimens 18, 19, 20, and 21 shows the degree of reproducibility that can be obtained with replicate tests. Since these tests were among the first to be run, the times at temperature (48 to 285 sec) prior to testing vary more than in subsequent tests. However, it is known that variations in exposure of

TABLE II.—SHORT-TIME TENSILE CREEP DATA FOR HK31A-H24[a] (LOT 51454).

Specimen	Test Temperature, deg Fahr	Stress, psi	Time at Temperature, sec	Strain Rate, in. per in. per min	Heating Rate, deg Fahr per sec	Loading Time, sec	Loading Strain, per cent	Per Cent Total Strain at Creep Times of:									Creep Time, sec, for Per Cent Creep Strain of:					
								Sec				Min										
								5	10	15	30	1	2	5	10	15	0.1	0.2	0.3	0.4	0.5	1.0
No. 101.....	500	8 000	15	0.0480	7.0	2.0	0.160	0.170	0.175	0.180	0.190	0.200	0.220	0.260	0.300	0.320	300	...	...	...	...	...
No. 102.....	500	12 000	15	0.0252	7.0	5.0	0.210	0.215	0.220	0.230	0.235	0.240	0.260	0.290	0.330	0.360	400	...	...	...	...	...
No. 103.....	500	14 000	15	0.0560	7.0	3.0	0.280	0.290	0.295	0.300	0.320	0.340	0.370	0.430	0.510	0.560	150	550	...	...	...	...
No. 104.....	500	16 000	15	0.0240	7.0	7.0	0.280	0.290	0.300	0.305	0.320	0.340	0.370	0.440	0.510	0.560	140	460	...	...	...	...
No. 105.....	500	18 000	15	0.0293	7.0	8.0	0.390	0.420	0.430	0.440	0.470	0.500	0.560	0.680	0.810	0.900	50	160	305	532	...	...
No. 106.....	500	20 000	15	0.1600	4.6	1.5	0.400	0.460	0.480	0.500	0.540	0.610	0.710	0.930	1.220	...	16	56	110	180	270	...
No. 107.....	500	21 000	15	0.1320	4.6	2.0	0.440	0.510	0.530	0.550	0.600	0.690	0.800	1.100	1.490	...	12	44	86	140	200	484
No. 108.....	500	22 000	15	0.0336	7.0	10.0	0.560	0.640	0.680	0.710	0.760	0.860	1.010	1.340	1.760	...	6	28	58	100	142	446
No. 109.....	500	23 000	15	0.0295	7.0	11.0	0.540	0.640	0.660	0.690	0.760	0.890	1.090	1.530	...	...	5	24	46	75	104	302
No. 110.....	500	24 000	15	0.3960	4.6	1.0	0.660	0.840	0.960	1.010	1.200	1.560	...	...	...	...	1	4	10	17	25	66
No. 111.....	600	5 000	15	0.0320	8.6	1.5	0.080	0.110	0.120	0.130	0.160	0.180	0.230	0.350	0.520	0.680	60	185	348	530	900	...
No. 112.....	600	8 000	15	0.0430	8.6	2.5	0.180	0.220	0.260	0.280	0.330	0.430	0.590	1.050	...	...	14	42	72	111	148	...
No. 113.....	600	10 000	15	0.0360	8.6	3.0	0.180	0.210	0.240	0.260	0.360	0.480	0.720	1.510	...	...	14	35	59	85	111	232
No. 114.....	600	12 000	15	0.0520	8.6	3.0	0.260	0.320	0.360	0.390	0.490	0.660	1.050	...	...	...	10	25	41	60	74	149
No. 115.....	600	14 000	15	0.0640	8.6	3.0	0.320	0.430	0.520	0.600	0.810	1.250	...	...	...	...	5	10	17	24	32	65

[a] Produced by process "B."

this order of magnitude have no significant effect on the properties of HK31A-H24. The variation in loading times (2 to 10 sec) appears to have no detrimental effects on the reproducibility of the data. However, it was noted that a loading time of 18 sec (as compared to 2 to 10 sec) did incur slightly higher loading strain and slightly lower creep strains to give approximately the same total extension figures.



NOTE.—T = tension; C = compression.
* R. B. Clapper, reference (**23**).

FIG. 6.—Comparison of 1-min Tension and Compression Isochronous-Stress - Strain Curves for HK31A-H24 Made by Process B to Tension-Isochronous Stress-Strain Curves for HK31A-H24 Made by Process A.

TABLE III.—MECHANICAL PROPERTIES[a] OF HK31A-H24 MAGNESIUM ALLOY.

Temperature, deg Fahr	Material Produced by Process B				Material Produced by Process A[b]		
	Elongation, per cent	Tensile Yield Strength, psi	Compressive Yield Strength, psi	Tensile Strength, psi	Elongation, per cent	Tensile Yield Strength,[a] psi	Tensile Strength, psi
75	10.0	32 700	28 600	40 700	14.0	27 500	35 500
300..........	...	28 400	26 400	31 400	...	23 400	25 500
400	...	25 800	25 300	28 400	...	20 700	22 200
500..........	...	22 300	22 400	26 200	...	17 400	19 500
600..........	...	9 800	10 100	18 000	...	6 200	13 500

[a] Elevated-temperature data obtained by resistance-heating method
[b] After Clapper (**23**).

Short-time tension creep data obtained on the same HK31A-H24 sheet at 500 and 600 F are given in Table II for comparison with the compression data. A comparison between the 1-min tension and compression isochronous stress-strain curves at 500 and 600 F is illustrated in Fig. 6 and shows the compressive strength to be somewhat lower than the tensile strength. This is in agreement with the finding of Yerkovich (25) who has noted the same behavior for other metals.

In 1958 Clapper (23) reported short-time tensile creep data for HK31A-H24 sheet produced by process A. Since this time a different process for producing HK31A-H24 has been developed, called process B. The new data reported in this paper have been obtained on material made by process B. In order to emphasize the magnitude of the difference in the short-time elevated-temperature properties between the two processes, some of Clapper's data obtained on material made by process A are plotted in Fig. 6 with some of the data from this research. It can be seen that the short-time creep strength of the material produced by process B is approximately 5000 psi higher than that produced by process A at both 500 and 600 F. In order to facilitate comparison of the two types of HK31A-H24, the properties of each of the materials are listed in Table III. It should be noted that the properties of the process B product given in this paper are not necessarily typical of current production but are representative of only the one lot tested.

SUMMARY AND CONCLUSIONS

The equipment described in this paper provides a means for obtaining compressive stress-strain data and compression creep properties of sheet alloys under conditions of rapid heating. This equipment is also satisfactory for determining Young's modulus in compression. The compression tests described here are more tedious and time consuming than equivalent tension tests (23) because of the problems of axial alignment, contact resistance and specimen support. Design data obtained with this equipment should be of particular interest to missile designers.

REFERENCES

(1) A. E. Flanigan, L. F. Tedsen, and J. E. Dorn, "Compressive Properties of Aluminum Alloy Sheet at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, pp. 951–969 (1946).

(2) G. J. Heimerl and W. M. Roberts, "Determination of Plate Compressive Strengths at Elevated Temperatures," *NACA Technical Note*, No. 1806, Nat. Advisory Committee for Aeronautics (1949).

(3) P. F. Barrett, "Compressive Properties of Titanium Sheet at Elevated Temperatures," *NACA Technical Note*, TN No. 2038, Nat. Advisory Committee for Aeronautics (1950).

(4) D. D. Doerr, "Compressive, Bearing, and Shear Properties of Several Nonferrous Structural Sheet Materials (Aluminum and Magnesium Alloys and Titanium) at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 52, pp. 1054–1078 (1952).

(5) J. Culmann, "Appareil pour la mesure de la résistance à l'écrasement sous charge à haute température (jusqu'à 1800 F)," *Métaux* (*Corrosion - Industries*), Vol. 28, No. 331, March, 1953, pp. 141–142.

(6) P. J. Hughes, J. E. Inge, and S. B. Prosser, "Tensile and Compressive Stress-Strain Properties of Some High-Strength Sheet Alloys at Elevated Temperatures," *NACA Technical Note*, TN No. 3315, Nat. Advisory Committee for Aeronautics (1954).

(7) D. E. Miller, "Determination of Physical Properties of Ferrous and Nonferrous Structural Sheet Materials at Elevated Temperatures," *Technical Report*, No. 6517, Wright Air Development Center, June, 1954.

(8) D. C. Hayward, "A Fixture for Testing Sheet Materials in Compression at Elevated Temperatures," *Technical Note*, MET. No. 193, Royal Aircraft Establishment, March, 1955.

(9) W. S. Hyler, "An Evaluation of Compression Testing Techniques for Determining Elevated Temperature Properties of Titanium Sheet," *Titanium Metallur-*

gical Laboratory Report, No. 43, Battelle Memorial Inst., Columbus, Ohio, June, 1956.

(10) F. J. Vawter, G. J. Guarnieri, L. A. Yerkovich, and G. Derrick, "Investigation of the Compressive, Bearing and Shear Creep-Rupture Properties of Aircraft Structural Metals and Joints at Elevated Temperatures," *Technical Report*, No. 54-270, Wright Air Development Center (1956–1958).

(11) D. E. Miller, "Determination of the Tensile, Compressive, and Bearing Properties of Ferrous and Nonferrous Structural Sheet Materials at Elevated Temperatures," *Technical Report*, No. 6517, Wright Air Development Center, Dec., 1957.

(12) B. L. Molander, C. R. Waldron, and J. C. Newland, "A Fixture for Compression Testing of Sheet Materials at Elevated Temperatures," ASTM BULLETIN, No. 225, Oct., 1957, pp. 37–39 (TP 187).

(13) R. L. Carlson and A. D. Schwope, "Investigation of Compressive-Creep Properties of Aluminum Columns at Elevated Temperatures," *Technical Report*, No. 52-251, Wright Air Development Center, Sept., 1952.

(14) A. H. Sully, "Creep Testing in Compression for Simple Creep Assessment," *Product Engineering*, April, 1953, pp. 150–153.

(15) N. P. Allen and W. E. Carrington, "Exploratory Creep Tests on Metals of High Melting Point," *Journal*, Inst. of Metals, July, 1954, pp. 525–533.

(16) E. E. Mothauser and D. Deveikis, "Preliminary Investigation of the Compressive Strength and Creep Lifetime of 2024-T3 Aluminum Alloy Plates at Elevated Temperature," *NACA Research Memorandum*, No. L55E11B, June, 1955.

(17) L. A. Yerkovich and G. J. Guarnieri, "Compression-Creep Properties of Several Metallic and Cermet Materials at High Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, pp. 732–754 (1955).

(18) E. C. Bernett, "Investigation of the Effects of Rapid Loading and Elevated Temperature on the Mechanical Behavior of Compressive and Column Members," Wright Air Development Center, Contract AF 33(616)-6043, Task 73605 (1959).

(19) T. C. McGill, J. V. Dutzi, and W. D. Ayers, "The Fluid Analogy to Aerodynamic Heating," Short-Time High Temperature Testing, Am. Soc. for Metals, Cleveland, Ohio (1958).

(20) J. N. Kotanchik, W. Woods, and R. A. Weinberger, "Investigation of Methods of Supporting Single-Thickness Specimens in a Fixture for Determination of Compression Stress-Strain Curves," *NACA Bulletin*, WRL-189 (Formerly RBL5E15), Nat. Advisory Committee for Aeronautics, May, 1945.

(21) D. A. Paul, F. M. Howell, and H. E. Grieshaber, "Comparison of Stress-Strain Curves Obtained by Single-Thickness and Pack Methods," *NACA Technical Note*, TN No. 819, Nat. Advisory Committee for Aeronautics, (1941).

(22) R. W. Fenn, Jr., "Determination of Young's Modulus Under Conditions of Relaxation," Papers on Metals, Am. Soc. Testing Mats. (1956). (Issued as separate publication *ASTM STP No. 196*.)

(23) R. B. Clapper, "Isochronous Stress-Strain Curves for Some Magnesium Alloys Showing Effects of Varying Exposure Times on Their Creep Resistance," *Proceedings*, Am. Soc. Testing Mats., pp. 812–825 (1958).

(24) R. W. Fenn, Jr. and J. A. Gusack, "Effect of Strain-Rate and Temperature on the Strength of Magnesium Alloys," *Proceedings*, Am. Soc. Testing Mats., pp. 685–696 (1958).

(25) L. A. Yerkovich, "Watch Out for Compression Loads," *Product Engineering*, Dec. 9, 1957.

DISCUSSION

Mr. J. R. Kattus.[1]—Has the author found a lower limit of clamping force below which he ran into buckling troubles in the sheet compression specimens?

Mr. R. W. Fenn, Jr. (*author*).—Buckling problems were encountered on tests at the lower temperatures. Since the buckling stress varies as a function of eccentricity of loading, Young's modulus (which changes with temperature) and the applied stress of the test, it is practically impossible to give a single value below which buckling would be encountered. However, it can be pointed out that using a clamping force of 40 lb (or a stress of 20 psi), 0.125 in. HK31A-H24 was tested successfully at 400 F and 26,000 psi in some cases, whereas buckling was encountered in others. At 500 F and 600 F no buckling was encountered with the same clamping force but lower applied test stresses.

Mr. R. B. Clapper[2] (*presented in written form*).—Because of the complexity of the problems attending a suitable test arrangement, it has been much easier for experimentalists to obtain tensile data and assume that the compression test data are equal. This practice has forced missile and space vehicle designers to use tensile data for modulus of elasticity, stress-strain curves, and isochronous stress-strain curves. Confirmation of the equality for these data for compression loading is lacking.

In his paper, the author has indicated that the problems of compression testing to high accuracy under rapid heating conditions is somewhat more severe than testing similarly in tension. He is unduly modest and restrained in his remark. The author is to be congratulated on his forthrightness to do what seems so rare among many recent investigators reporting on their work. He admitted that he had had some problems, and proceeded to discuss them frankly. In a paper on methods which other investigators would like to follow, it is important to know where one can go wrong as well as to know what works well.

In the written text the author mentioned that magnesium alloys are not recommended for salt bath solutions, and oil baths have an upper limit of about 600 F useful temperature range. We have recently initiated some work using a low-melting eutectic alloy. It was found that cerro-matrix is not corrosive to magnesium-thorium alloys and was useful for short times, up to about 900 F for 10 min. We think immersion baths are preferable in certain rapid-heating tests to resistance heating, if suitable baths are available in the required temperature range.

I would like to ask the author to discuss the problems expected during testing in a similar manner for other materials, such as beryllium, super steels, and refractory metals, regarding the control of temperature gradients through the specimen when using resistance heating.

Mr. Fenn.—In answer to Mr. Clapper's question on testing materials other than magnesium, I would expect the test techniques described in this paper to be applicable if the specimen supporting jig could be made from a material which has significantly higher electrical resistivity than the test specimen. This would minimize shunting of the electric current around the test specimen.

---

[1] Southern Research Inst., Metallurgy Division, Birmingham, Ala.

[2] Structural Engineer, Missile and Space Division, Lockheed Aircraft Co., Sunnyvale, Calif.

MR. E. C. BERNETT[3] (*presented in written form*).—The design of safe minimum weight missile structures is a very complex problem, especially when rapid changes in temperature and stresses are involved. Where compression loads are critical, the problem is even more difficult because compression data are relatively scarce. Tests such as described here by Mr. Fenn are of great value, and other suppliers of structural materials should be urged to support similar research.

Relative to future work in the field of compression testing, much remains to be done with regard to improving test equipment so that thorough evaluations can be carried out on all the useful structural materials. The test temperatures must be extended well beyond those described in the subject paper. The test procedures should be standardized so that the data from different laboratories may be compared directly. Regarding equipment, the specific problems that must be resolved are discussed below:

1. The correct lateral support loads to be used on each material at each temperature should be established. To minimize frictional errors it would probably be best to "couple" the lateral support load to the principal (compression) load. The lateral support load could then start off at a low level and increase proportionately as the compression load increased.

2. In all compression tests, particularly at elevated temperatures, unknown errors due to galling, local buckling and jamming, etc., can occur. To account for this, provision should be made to measure and record the compression load on both sides of the lateral support fixture. The difference in these two readings would provide some indication of frictional errors encountered in each test.

3. Equipment for measuring, recording, and controlling of temperature in this type of test setup requires major improvement. The rapid heating rates involved and the fact that the lateral support fixture is in contact with portions of the specimen are two complicating features. Accurate temperature-measuring equipment is needed to establish the temperature along the full test portion at all times during the test.

4. The design and finish machined accuracy of the test specimens, the rigidity and alignment of the test equipment, and the accuracy of the strain measuring device are other factors to be considered.

MR. FENN.—A system of coupling the lateral support load to the principal compression load as described by Mr. Bernett would be desirable if it could be done conveniently. The use of currently available recording equipment with the specimen-supporting jig as described in the paper would permit continuous recording of lateral support loads. I cannot agree with Mr. Bernett that equipment for measuring, recording, and controlling temperature in the subject type of tests is in need of major improvement. I think the equipment used in this investigation is satisfactory and that even better systems are commercially available. However, I do agree that the rigidity and alignment of the test equipment are important items to be considered in compression testing of sheet.

---

[3] Assistant Supervisor, Materials and Process Section, The Marquardt Corp., Van Nuys, Calif.

PRELIMINARY EVALUATION OF THERMOCOUPLE ATTACHMENT METHODS FOR RADIANT-HEATING APPLICATIONS*

By Jay F. Burcham[1]

Synopsis

A simple and economical thermocouple installation method was required for accurately sensing the temperature of a metallic surface exposed to radiant heating. For evaluation purposes, alumel-chromel thermocouples were installed on 0.050-in. thick steel panels, using eight methods. The test panels were exposed to radiant heating on one side. The radiant heat input was controlled to cause the temperature of the panels to increase at nominal rates of 50 F and 100 F per sec to 1500 F. The approximate accuracy of each type of installation was determined by comparing the measured and calculated temperature gradients through the thickness of each panel. The eight types of installations are evaluated primarily on the basis of accuracy; but reliability, ease of installation, and surface continuity effects are also considered. The approximate accuracy of each type of installation under the conditions investigated is presented. Three of the installations investigated should not be considered further for radiant heating environments because of poor accuracy characteristics; the accuracy of two other installations was marginal. The accuracy of each installation is dependent upon many factors.

When testing materials intended for structural applications in high-speed aircraft and missiles, it is often necessary to simulate the environmental conditions produced by aerodynamic heating. This requires the generation of high heating rates. Radiant heating methods are sometimes used for this purpose. Measurement and control of the heat fluxes imposed are usually dependent upon the accurate measurement of temperature. For many applications the most convenient, economical, and dependable method of measuring temperatures is by use of thermocouples. Unfortunately, the accuracy of many thermocouple installations, suitable for steady-state temperature measurements, is adversely affected when exposed to radiant heating. The object of this investigation was to establish and evaluate methods by which the temperature of a metallic surface subjected to radiant heating could be accurately measured by thermoelectric means.

Several factors must be considered when evaluating thermocouple application methods. Accuracy of temperature measurement is of primary importance, but other characteristics such as reliability, ease of installation, cost, and the effect of the thermocouple on the over-all test must be considered. The evaluation of a particular thermocouple installation can be influenced by many variables, the

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Structures Engineer, Satellite Systems Test Services, Lockheed Aircraft Corp., Missiles and Space Division, Sunnyvale, Calif.

most important of which are: the heating rate, the temperature range, the surrounding medium, the test material type and size, and the thermocouple wire material, type, size, and insulation.

A comprehensive investigation of all of the factors affecting the performance of a thermocouple installation could not be undertaken. The scope of the investigation was limited to securing information on thermocouple installations which would be applicable to current and scheduled radiant-heat tests of materials and structures. A literature search failed to disclose any information on the relative importance of the various factors which can influence thermocouple performance. A set of test factors was established for the investigation. All factors were kept constant except the method of installation.

Eight methods of installing thermocouples were selected, which included those being used at the time of the investigation and modifications designed to improve undesirable reliability characteristics of two installations. Test installations were mounted on one side of a steel test panel. On the opposite side of the panel comparison thermocouples were installed. The integrity of all of the installations was tested at low temperatures of known value. Then the side of the test panel containing the test installations was subjected to two different radiant-heating programs using infrared lamps as the heat source. The accuracy of the test thermocouple installations was determined by comparing the measured and calculated temperature gradients through the thickness of the test panel.

PROCEDURE

Test Specimens:

The test specimens were 0.050 by 3.0 by 6.0-in. panels of normalized type 4130 steel on which were installed 17 thermocouples. Type 4130 steel was selected as the test material because it was a common material of known thermal properties and was representative of the types of materials being tested under radiant-heat conditions at the time of this investigation. The configuration of the panels and locations of thermocouples are shown in Fig. 1. A single panel was used to evaluate two methods of attachment. On the top surface of the panel, which was the surface exposed directly to radiant heating, two types of thermocouples were located alternately at 0.50 in. intervals along the perimeter of a 1.00-in. square placed centrally on the panel. Eight additional thermocouples were mounted on the bottom surface of the panel in a corresponding 1.00-in. square arrangement which was displaced 0.12 in. along the longitudinal axis of the panel relative to the square on the top surface. The 0.12-in. offset was required to avoid interference between some types of corresponding top and bottom installations. An offset greater

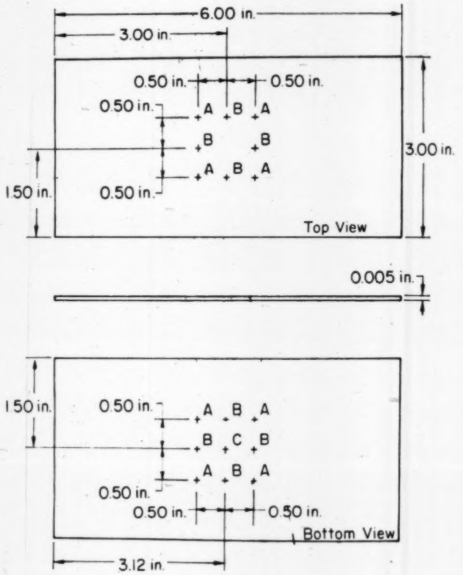


FIG. 1.—Thermocouple Evaluation Test Panel Configuration.

than 0.12 in. was not considered desirable because of minor variations in heat input which occur over the surface of a specimen directly exposed to radiant heaters using quartz lamps. An additional thermocouple was mounted on the bottom surface for controlling the rate of temperature rise in the panel.

Thermocouple Installations:

No. 28 gage chromel-alumel thermocouple wire of solid, parallel, duplex construction with fiberglass insulation was used for all installations. The thermocouple wire used was selected for several reasons. Chromel-alumel is generally recognized as the best combination of thermoelements for the 70 F to 1500 F temperature range being investigated because the emf per degree response of the couple is relatively high and essentially linear, and its resistance to oxidation is good. Rare-metal couples such as the platinum-rhodium types have superior oxidation resistance but have a low response and are expensive to use. Other common thermoelement combinations such as copper-constantan and iron-constantan are limited to maximum temperatures of 600 and 1300 F respectively because of excessive oxidation at higher temperatures. No. 28 gage was selected as the best wire-size compromise. A smaller wire size would decrease undesirable mass and surface discontinuity effects but would not be mechanically reliable when subjected to normal test laboratory installation and handling conditions. The experiences of previous test programs demonstrated the durability of fiberglass as an insulating material when subjected to heating and high temperatures. The insulation of the thermocouple wire which was directly exposed to the radiant heat was painted with a silver-colored metallic paint to decrease the amount of radiant energy absorbed by the insulation.

The thermocouple installations used in the subject investigation were supported mechanically by forming a half-loop in the wire as close to the measuring junction as the installation instructions permitted, placing thin anchoring strips of stainless steel feeler stock over the insulated wire after the loop, and spot-welding the feeler stock directly to the surface of the test specimen on each side of the wire.

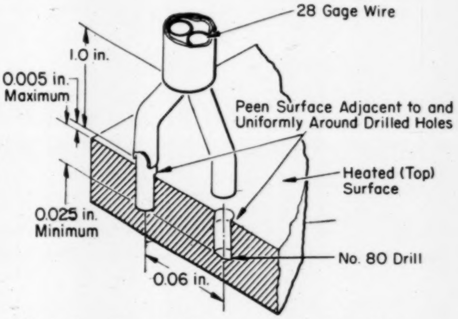

28 Gage Wire
1.0 in.
0.005 in. Maximum
Peen Surface Adjacent to and Uniformly Around Drilled Holes
Heated (Top) Surface
0.025 in. Minimum
No. 80 Drill
0.06 in.

FIG. 2.—Type I Thermocouple Installation—Each Lead Staked Individually.

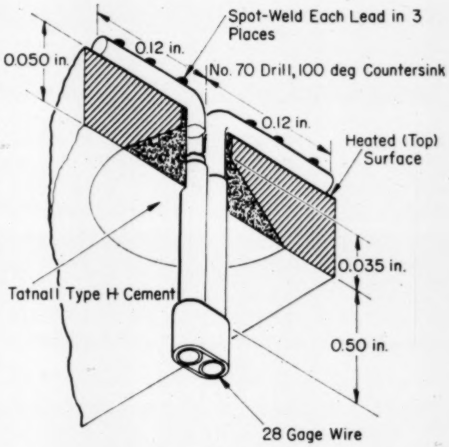

Spot-Weld Each Lead in 3 Places
0.12 in.
0.050 in.
No. 70 Drill, 100 deg Countersink
0.12 in.
Heated (Top) Surface
0.035 in.
Tatnall Type H Cement
0.50 in.
28 Gage Wire

FIG. 3.—Type II Thermocouple Installation—Each Lead Spotwelded Individually with Countersunk Cemented Attachment.

The design parameters for each method of installation were established before and carefully adhered to during installation of the thermocouples. The installation instructions are given in the following paragraphs. The designation top surface refers in all cases to the surface which will be exposed to the radiant heat.

The type I installation (each lead staked individually) is shown schematically in Fig. 2. Prepare the thermocouple wire by stripping back the outside insulation 1.0 in. and stripping back the insulation on each individual lead about 0.030 in. On the top surface of the specimen, drill two No. 80 holes 0.06 in. apart to a minimum depth of 0.025 in. The drilled hole must not penetrate the bottom surface. Place each thermocouple lead in a separate hole. The minimum amount of lead engagement should be 0.025 in. and maximum length of exposed lead should be 0.005 in. Stake the leads in place by carefully peening the top surface adjacent to and uniformly around the drilled holes. Pull gently on the staked leads to determine if they are mechanically secured. Support the leads as close to the junction as possible, considering thermal and load effects.

The type II installation (each lead spotwelded individually with countersunk cemented attachment) is shown schematically in Fig. 3. The installation instructions are applicable only for 0.050-in. thick material. Prepare the thermocouple wire by stripping back the outside insulation 0.5 in. and stripping back the insulation on each individual lead 0.15 in. Drill a No. 70 hole through the specimen. Countersink the bottom surface 0.350 in. deep by 100 deg. Place both leads in the hole so that 0.125 in. of each lead projects from the top surface. Bend over and spotweld each lead to the top surface, individually. The bare leads must not touch each other or the specimen, except at the top surface. Fill the countersink and drilled hole with Tatnall type H cement. The cement should be flush with both the top and bottom surfaces. Completely cure the cement, preferably reaching the maximum test temperature at least once before using the thermocouple installation.

The type III installation (insulation and bare lead staked) is shown schematically in Fig. 4. Prepare the thermocouple wire by stripping back the outer insulation 0.5 in. more than the thickness of the specimen and stripping back the insulation on each individual lead 0.10 in. From the bottom surface, drill two No. 64 holes 0.06 in. apart to within 0.005 in. of the top surface. Drill through to the top surface with a No. 80 drill. Place each lead in a separate hole with the insulation resting on the bottom of the No. 64 hole and the bare lead projecting through the No. 80 hole. Stake the bare

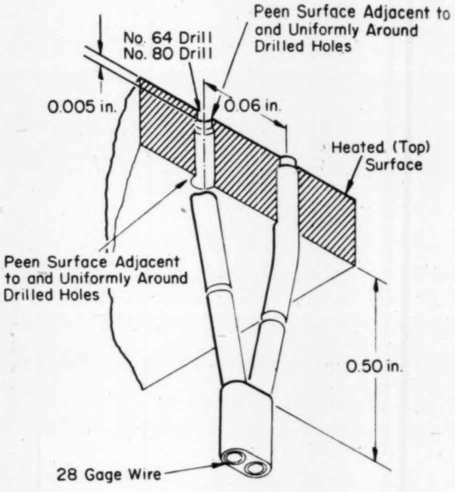

Peen Surface Adjacent to and Uniformly Around Drilled Holes
No. 64 Drill
No. 80 Drill
0.005 in.
0.06 in.
Heated (Top) Surface
Peen Surface Adjacent to and Uniformly Around Drilled Holes
0.50 in.
28 Gage Wire

FIG. 4.—Type III Thermocouple Installation—Insulation and Bare Lead Staked.

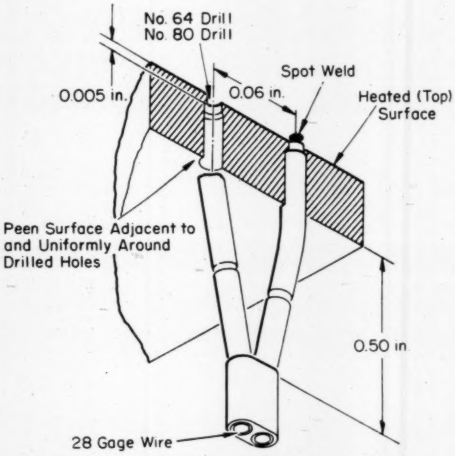

No. 64 Drill
No. 80 Drill
Spot Weld
0.005 in.
0.06 in.
Heated (Top) Surface
Peen Surface Adjacent to and Uniformly Around Drilled Holes
0.50 in.
28 Gage Wire

FIG. 5.—Type IV Thermocouple Installation—Insulation Staked and Bare Lead Spotwelded.

leads in place by carefully peening the top surface adjacent to and uniformly around the drilled holes. Pull gently on the staked leads to determine if they are mechanically secured. Stake the insulation in place by peening around the No. 64 drilled holes on the bottom surface. Clip off the exposed excess bare leads flush with the top surface.

The type IV installation (insulation staked and bare lead spotwelded) is shown schematically in Fig. 5. Prepare the thermocouple wire by stripping back the outer insulation 0.5 in. more than the thickness of the specimen and stripping back the insulation on each individual lead 0.10 in. From the bottom surface, drill two No. 64 holes 0.06 in. apart to within 0.005 in. of the top surface. Drill through to the top surface with a No. 80 drill. Place each lead in a separate hole with the insulation resting on the bottom of the No. 64 hole and the bare lead projecting through the No. 80 hole. Spotweld the bare leads to the top surface of the specimen. Pull gently on the spotwelded leads to determine if they are mechanically secured. Stake the insulation in place by peening around the No. 64 drilled holes on the bottom surface. Clip off the exposed (excess) bare leads flush with the top surface.

The type V installation (each lead spotwelded individually) is shown schematically in Fig. 6. Prepare the thermocouple wire by stripping back the outer insulation 1.0 in. and stripping back the insulation on each individual lead 0.12 in. Spotweld the bare leads to the top surface with the leads parallel and 0.06 in. apart. Spotweld each lead in three places to increase mechanical reliability. A minimum of 0.25 in. of insulated lead should extend along the top surface. No bends should occur within 0.25 in. of the end of the insulation portion of each individual lead. Mechanically support the leads if mechanical and thermal conditions permit.

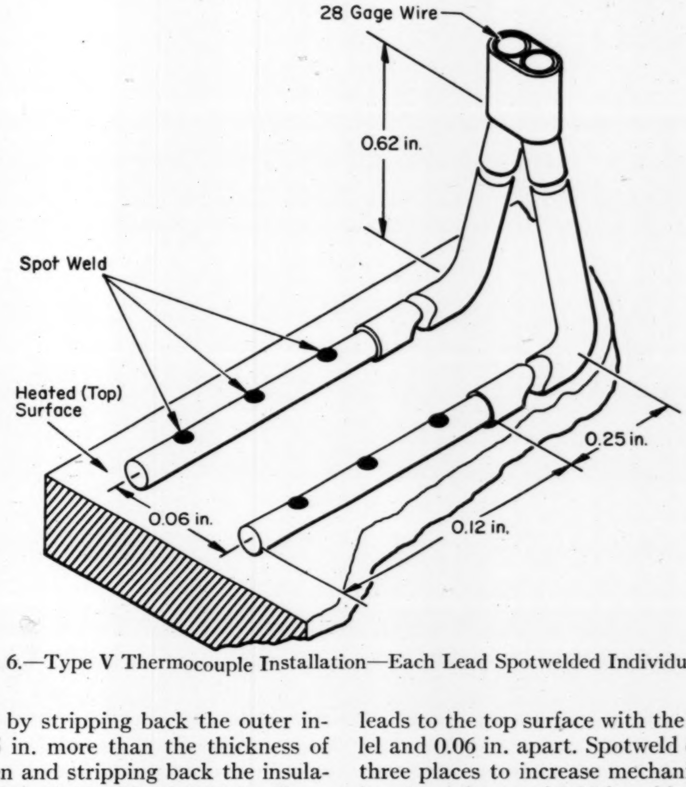


FIG. 6.—Type V Thermocouple Installation—Each Lead Spotwelded Individually.

The type VI installation (leads twisted and staked) is shown schematically in Fig. 7. Prepare the thermocouple wire by stripping back the outer insulation 0.50 in. and strip·

ping back the insulation on each individual lead 0.25 in. Twist the bare leads together continuing the twist to the individual lead insulation. Cut off the twisted portion to a length of 0.030 in. and spotweld in three places. Drill a No. 71 hole at least 0.030-in. deep from the top surface. Drilled hole must not penetrate bottom surface. Insert the twisted thermocouple in the hole. The minimum amount of twisted lead engagement should be 0.030 in. and the maximum length of exposed lead should be 0.005 in. Stake the twisted leads in place by carefully peening the top surface adjacent to and uniformly around the drilled holes. Pull gently on the staked leads to determine whether or not they are mechanically secured.

The type VII installation (leads twisted and spotwelded) is shown schematically in Fig. 8. Prepare the thermocouple wire by stripping back the outer insulation 0.75 in. and stripping back the insulation on each individual lead 0.25 in. Twist the bare leads together continuing the twist 0.25 in. past the bare portion of the leads. Spotweld the twisted bare leads to the top surface of the test specimen in three places. The twisted portion of the insulation covered leads should extend along the top surface of the test specimen. No bends should occur within 0.25 in. of the end of the insulated portion of the leads.

The type VIII installation (leads crossed and spotwelded) is shown schematically in

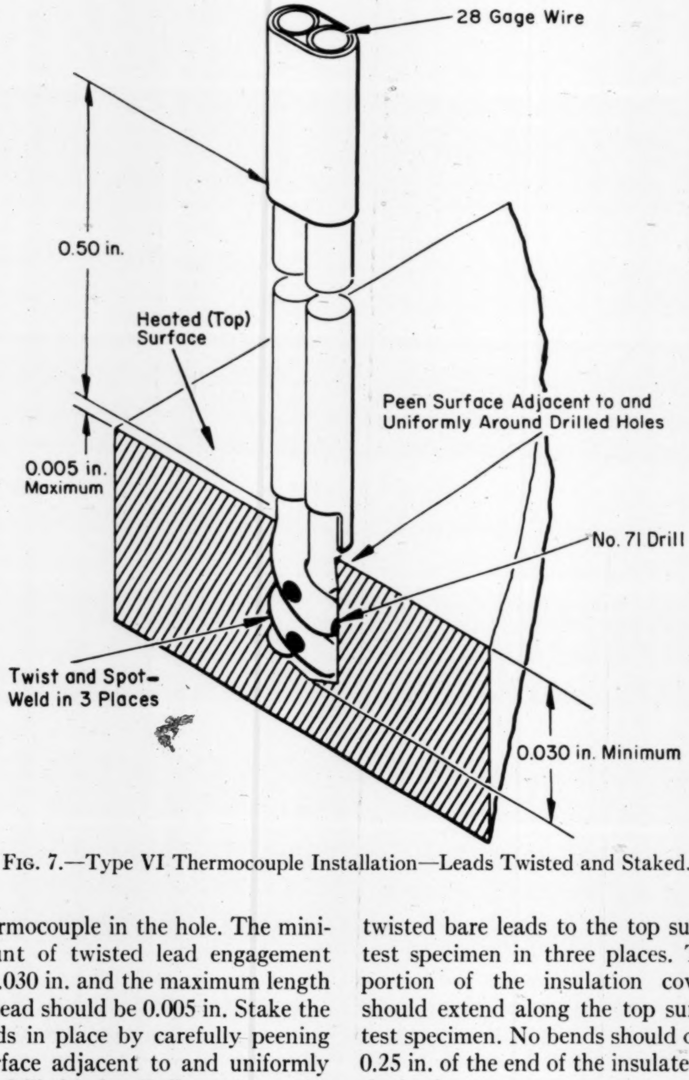

28 Gage Wire
0.50 in.
Heated (Top) Surface
Peen Surface Adjacent to and Uniformly Around Drilled Holes
0.005 in. Maximum
No. 71 Drill
Twist and Spot-Weld in 3 Places
0.030 in. Minimum

FIG. 7.—Type VI Thermocouple Installation—Leads Twisted and Staked.

Fig. 9. Prepare the thermocouple wire by stripping back the outer insulation 0.75 in. and stripping back the insulation on each individual lead 0.12 in. Cross the bare leads

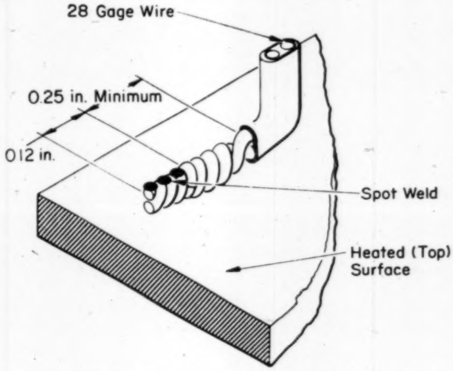


FIG. 8.—Type VII Thermocouple Installation—Leads Twisted and Spotwelded.

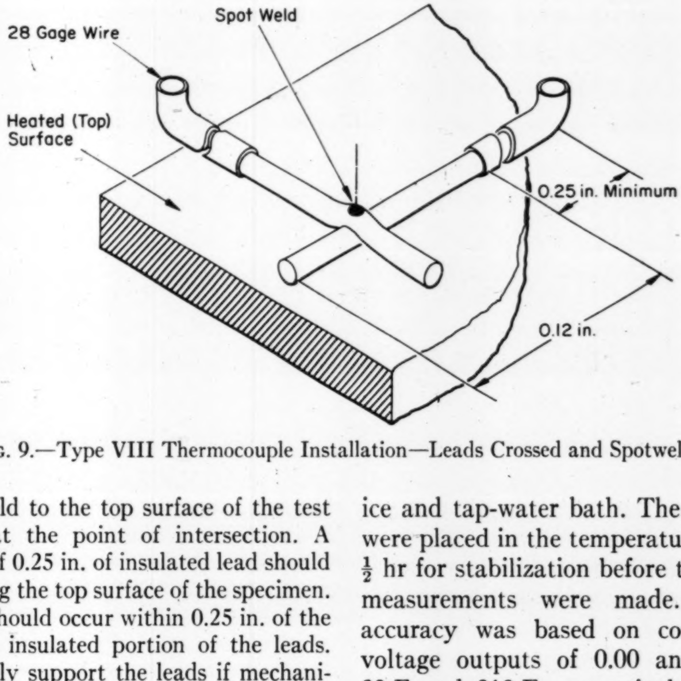


FIG. 9.—Type VIII Thermocouple Installation—Leads Crossed and Spotwelded.

and spotweld to the top surface of the test specimen at the point of intersection. A minimum of 0.25 in. of insulated lead should extend along the top surface of the specimen. No bends should occur within 0.25 in. of the end of the insulated portion of the leads. Mechanically support the leads if mechanical and thermal considerations permit.

Steady State Temperature Evaluation:

The thermocouple installations were tested to determine their accuracy at the standard temperatures of 32 F and 212 F. In addition to establishing basic accuracy, the tests were used to detect installations having open or reversed leads. The accuracy of the installations under steady state conditions at higher temperatures was not determined because the test panels could not be accommodated within an available thermocouple calibration furnace. Subsequent calibration tests of the type of thermocouple wire used confirmed the manufacturer's guaranteed accuracy of ±2 F from 0 to 530 F and $\pm\frac{3}{8}$ per cent from 530 to 2300 F.

Crushed ice and tap water and boiling tap-water baths were used to establish the standard temperatures of 32 F and 212 F, respectively. A reference temperature of 32 F was established by a crushed-ice and tap-water bath. The test panels were placed in the temperature baths for $\frac{1}{2}$ hr for stabilization before temperature measurements were made. Indicated accuracy was based on correct millivoltage outputs of 0.00 and 4.098 at 32 F and 212 F, respectively, as determined from reference (**1**)[2] by the methods of Appendix I.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Radiant-Heating Evaluation:

The thermocouple installations were directly exposed to radiant heating in order to determine its effects on the accuracy and reliability of the various methods of attachment being investigated.

The test apparatus used for the radiant-heating evaluation of each test panel is shown in Fig. 10. The test panel was held in a horizontal position by a holder which supported it along its 6-in. edges. A radiant heater was located directly above the test panel. The radiant heater used type 2000T3 infrared lamps. The heater subjected the test thermocouple installations on the top surface of the panel to direct radiant-heat fluxes which caused the panel temperatures to increase as shown in Fig. 11(*a*). The panel shielded the comparison thermocouple installations on its bottom surface from exposure to the radiant-heat flux.

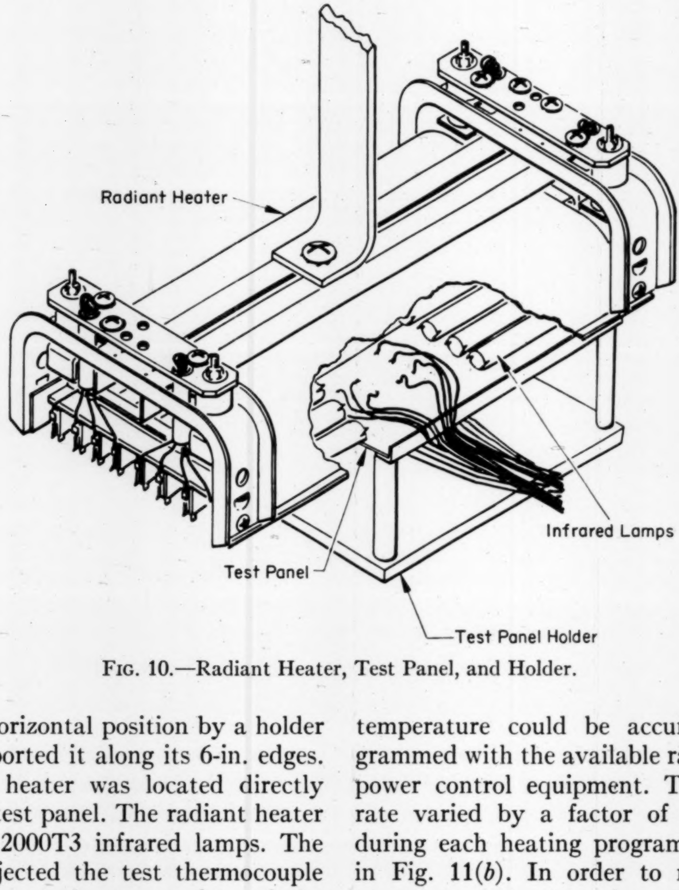


FIG. 10.—Radiant Heater, Test Panel, and Holder.

The test panels were first subjected to a controlled heat flux which produced an increase in panel temperature from 70 to 1500 F at a constant rate of 50 F per sec. The panels were then subjected to a controlled heat flux which produced an increase in panel temperature from 70 to 1500 F at a constant rate of 100 F per sec. Heating control was based on a constant temperature rise rate because temperature could be accurately programmed with the available radiant-heat power control equipment. The heating rate varied by a factor of about two during each heating program as shown in Fig. 11(*b*). In order to maintain a constant temperature rise rate the heating rate had to be varied to compensate for the changes which occurred in the thermal properties of the steel panel material as panel temperature increased. The regulation characteristics of the radiant-heat controller caused momentary surges in heating. The surges produced instantaneous heating rates which were about three times as high as the mean heating rates. The surges were of short duration. Test panel temperatures used for control purposes were sensed by a type I thermocouple installation (each lead staked individually) attached to the bottom surface of the panel.

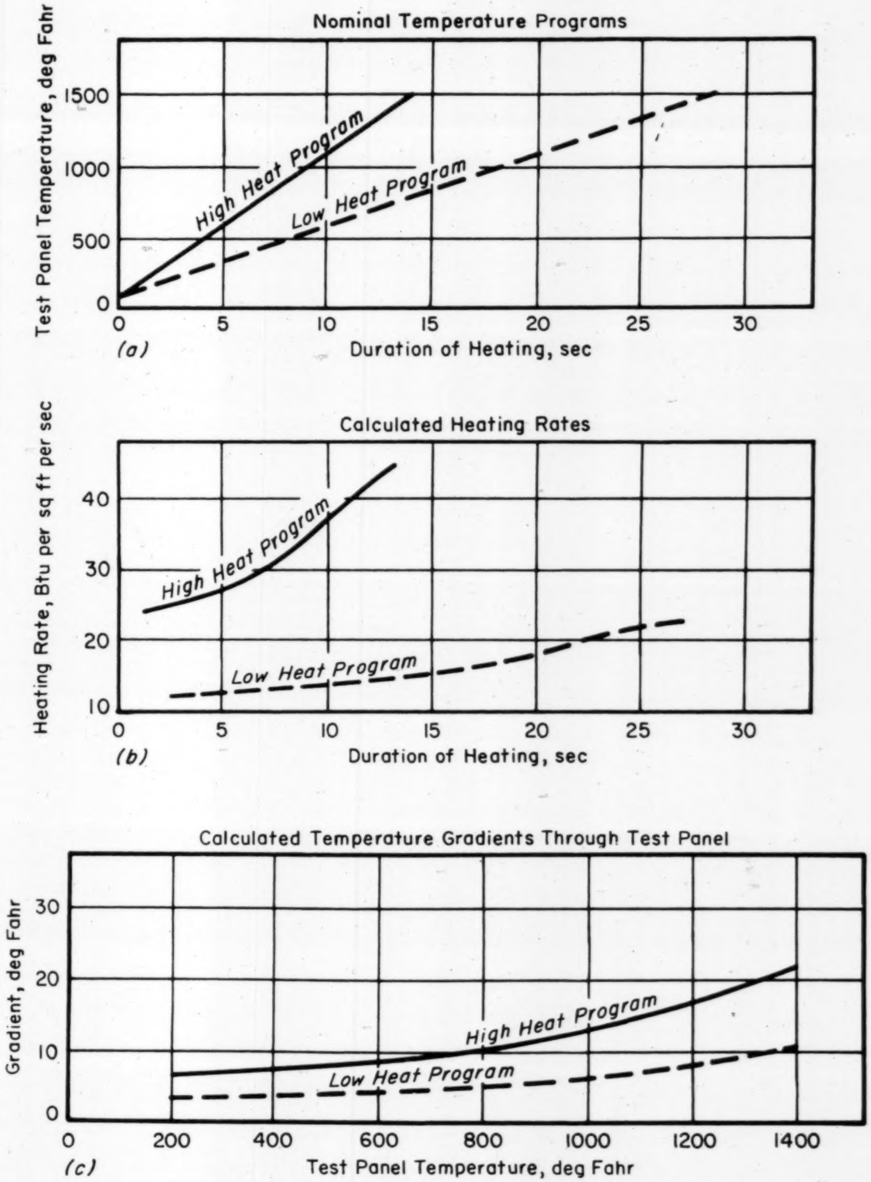


FIG. 11.—Radiant-Heating Evaluation Thermal Conditions.

The millivoltage signals from each thermocouple installation were monitored continuously during each heating program by a recording oscillograph. Oscillograph calibration was accomplished by imposing known millivoltages on each temperature monitoring circuit and recording the resulting oscillograph galvanometer deflections. Over-all measuring accuracy was ±10 F or ±1 per cent of the indicated temperature, whichever is greater, which included the effects of thermocouple wire inaccuracy, monitoring system inaccuracy, and chart resolution and readability.

The accuracy of each thermocouple installation was determined by comparing the temperature indications of the test thermocouple installation on the exposed top surface with the indication of the corresponding comparison thermocouple on the bottom surface. The accuracy was considered to be the difference in the indications of the test and comparison thermocouple installations less the calculated temperature gradient through the panel. For the purpose of determining accuracy, the indications of the comparison thermocouple installations on the bottom surface were considered correct.

The temperature gradients which existed between the top (heated) surface and bottom surface of the test panel were approximately those shown in Fig. 11(*c*). The method used to calculate the gradients and the simplifying assumptions which were made are given in Appendix II.

RESULTS

Table I summarizes the results obtained for each type of thermocouple installation tested in terms of accuracy, ease of installation, mechanical durability, and local effects.

TABLE I.—GENERAL EVALUATION OF THERMOCOUPLE INSTALLATIONS.

Thermocouple Installation		Characteristics			
Type	Description	Accuracy	Installation[a]	Mechanical Durability[b]	Absence of Local Effects[c]
I........	Each lead staked individually	good	poor	fair	good
II.......	Each lead spotwelded individually with countersunk cemented attachment	good	poor	good	poor
III......	Insulation and bare lead staked	excellent	fair	excellent	fair
IV......	Insulation staked, bare lead spotwelded	fair	fair	excellent	fair
V.......	Each lead spotwelded individually	fair	excellent	poor	excellent
VI......	Leads twisted and staked	poor	good	good	fair
VII.....	Leads twisted and spotwelded	poor	good	fair	excellent
VIII....	Leads crossed and spotwelded	poor	excellent	poor	excellent

[a] Ease and economy of installation by semi-skilled laboratory technicians.

[b] Ability of the installation to withstand normal handling.

[c] Introduction of undesirable discontinuities and heat sinks.

All of the thermocouple installations were functioning at the conclusion of five or more temperature cycles to 1500 F.

All of the thermocouple installations were accurate within the limits of ± 2F guaranteed for the thermocouple wire by its manufacturer at temperatures of 32 and 212 F.

The approximate accuracies of each type of thermocouple installation, when subjected to radiant heating, are shown in Figs. 12 and 13. The results shown are those of only one of the installations of each type tested. The results are representative of the performance of all identical installations tested within the testing accuracy of ±10 F or ±1 per cent of the indicated temperature, whichever is greater.

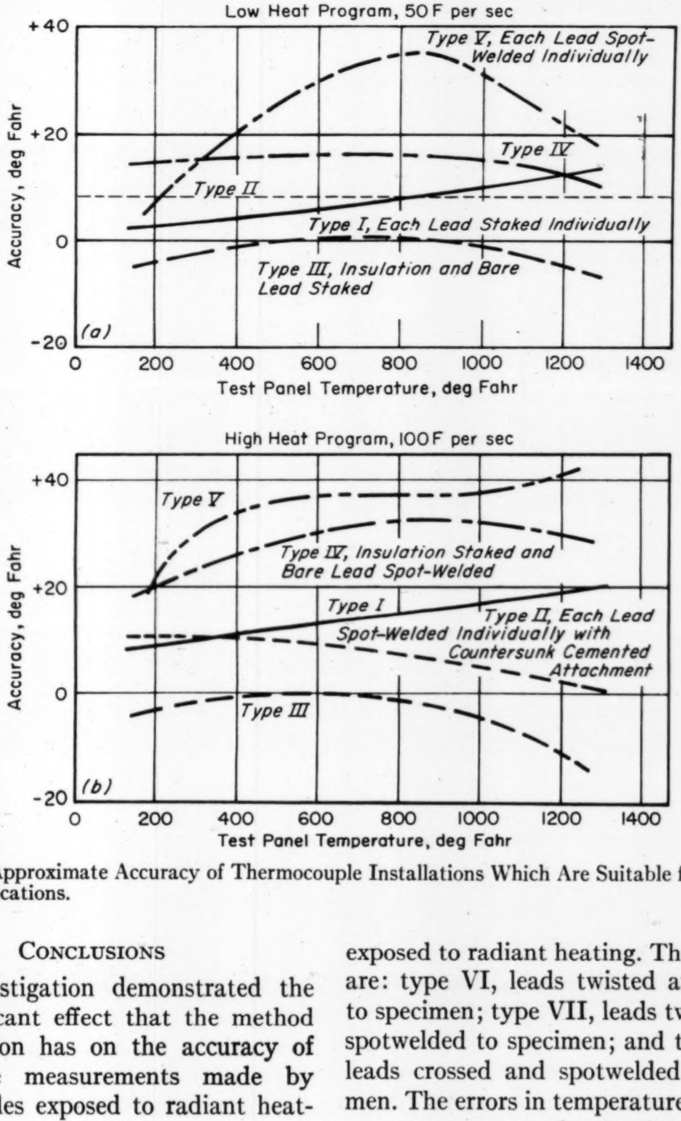


FIG. 12.—Approximate Accuracy of Thermocouple Installations Which Are Suitable for Radiant-Heating Applications.

CONCLUSIONS

The investigation demonstrated the very significant effect that the method of installation has on the accuracy of temperature measurements made by thermocouples exposed to radiant heating. Methods of installation which can be used for steady state temperature measurements often cannot be used when subjected to radiant heating because they do not give accurate results.

Three methods of installation which were investigated are not recommended for temperature measurements in regions exposed to radiant heating. The methods are: type VI, leads twisted and staked to specimen; type VII, leads twisted and spotwelded to specimen; and type VIII, leads crossed and spotwelded to specimen. The errors in temperature measurement were so great for the three methods listed that it is not considered likely the accuracy would improve to within acceptable limits if any of the following parameters were changed: temperature range; surrounding medium; test material type and size; and thermocouple wire material, type, size and insulation. Only a reduction in the heating rate to essentially a steady state condition would result in acceptable accuracy qualities.

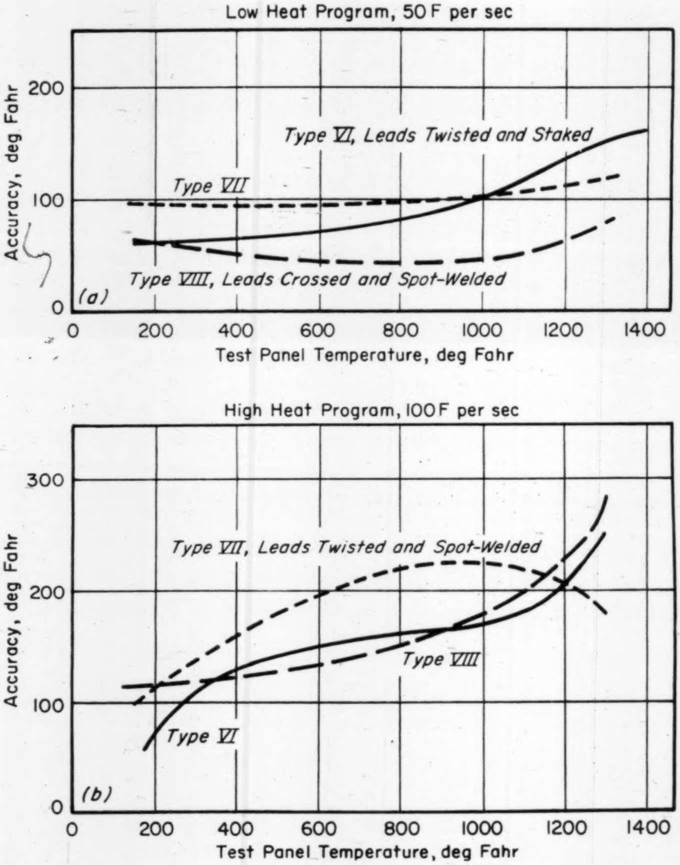


FIG. 13.—Approximate Accuracy of Thermocouple Installations Not Suitable for Radiant-Heating Applications.

The other five methods of installation investigated exhibited acceptable performance characteristics for use when exposed to radiant heating. For conditions other than those investigated, a particular installation must be used with caution because the effects of variables other than method of installation on accuracy have not been adequately determined. The results of this investigation will serve as a useful guide in the selection of a method of installation for a particular application. It is recommended that the accuracies which are reported only be used as approximations. For each particular application, the installation selected for use should be subjected to evaluation tests following the procedure used in this investigation.

This investigation was preliminary in nature and severely limited in scope. It should serve as the basis for additional programs of investigation which will examine other methods of installations and the effects of varying the test parameters held constant in this investigation.

REFERENCES

(1) Minneapolis-Honeywell Regulator Co., "Conversion Tables for Brown Pyrometers," *Service Manual*, No. 59095M, Issue 1.

(2) Dike, "Thermoelectric Thermometry," Leeds & Northrup Co. (1958).

(3) M. Jakob and G. A. Hawkins, "Elements of Heat Transfer and Insulation," John Wiley & Sons, Inc., New York, N. Y., 2nd Edition (1952).

(4) Baker, Ryder, and Baker, "Temperature Measurement in Engineering," John Wiley & Sons, Inc., New York, N. Y., Vol. 1 (1953).

APPENDIX I

CALCULATIONS OF INDICATED ERRORS-STEADY STATE LOW-TEMPERATURE EVALUATION

A. *Error at Ice Point:* Reference and measuring junctions in same crushed-ice and water bath.

at 32 F, mv output = 0 because 0 temperature difference exists between junctions

From reference (1):

30 F = −0.04 mv therefore −1 F = −0.02 mv

35 F = +0.07 mv therefore +1 F = +0.023 mv

B. *Error at Boiling-Water Point:* Reference junction in crushed-ice and water bath. Measuring junction in boiling-water bath.

From reference (1):

210 F = 4.05 mv
215 F = 4.17 mv

5 F = 0.12 mv therefore ±1 F = ±0.024 mv

at 212 F, mv output = 4.05 + 2(0.024) = 4.098 mv

APPENDIX II

CALCULATION OF TEMPERATURE GRADIENT THROUGH 0.050-IN. THICK 4130 STEEL

In order to determine the approximate temperature difference existing between the top (heated) and bottom surfaces of the test panel, two assumptions were made. Considering the thin cross-section of 0.050 in. and the small temperature gradient which exists, the following assumptions can be made:

$\frac{\partial T}{\partial t}$ is a constant throughout the thickness of the test panel at any time t

By neglecting the convective and radiant-heat losses from the bottom surface, which under the worst conditions (test panel at 1400 F and surrounding environment at 100 F) amounts to only about 10 per cent of the total heat input to the panel, the following assumption can be made:

$\frac{dT}{dx} = 0$ at the bottom surface of the panel

The basic equation for the transfer of heat by conduction is

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial x^2}$$

where:

T = temperature of panel, deg Fahr,
t = time, sec,
x = distance through panel, ft, and
α = thermal diffusivity, sq ft per sec.

Rearranging terms and applying the first assumption given above

$$\frac{d^2T}{dx^2} = \frac{1}{\alpha}\frac{dT}{dt}$$

Integrating with respect to x

$$\frac{dT}{dx} = \frac{1}{\alpha}\frac{dT}{dt}x + C_1$$

Applying the second assumption given above the constant C_1 can be determined at $x = a$, $\frac{dT}{dt} = 0$ where a = thickness of test panel

$$C_1 = -\frac{1}{\alpha}\frac{dT}{dt}a$$

and

$$\frac{dT}{dx} = \frac{1}{\alpha}\frac{dT}{dt}[x - a]$$

Integrating with respect to x over the entire thickness of the panel

$$T_2 - T_1 = \frac{1}{\alpha}\frac{dT}{dt}\left[\frac{-a^2}{2}\right]$$

or

$$T_1 - T_2 = \frac{1}{\alpha}\frac{dT}{dt}\left[\frac{a^2}{2}\right]$$

where:

T_1 = temperature of top (heated) surface, deg Fahr.

For the specific conditions of $\frac{dT}{dt} = 50$ F per sec and $a = 0.050$ in.

$$T_1 - T_2 = \frac{50\,(0.050)^2}{2\,(144)\alpha}$$

$$T_1 - T_2 = \frac{4.34 \cdot 10^{-4}}{\alpha}$$

For the specific conditions of $\frac{dT}{dt} = 100$ F per sec and $a = 0.050$ in.

$$T_1 - T_2 = \frac{8.68 \cdot 10^{-4}}{\alpha}$$

Table II contains the thermal properties of type 4130 steel at various temperatures which were used to calculate the gradient through the 0.050-in. thick test panel. The calculated gradients for temperature rise rates of 50 F per sec and 100 F per sec are shown in Fig. 11(*c*).

TABLE II.—THERMAL PROPERTIES OF TYPE 4130 STEEL.

Nominal Temperature, deg Fahr	Thermal Conductivity, Btu ft per sec deg Fahr	Specific Heat, Btu per lb per deg Fahr	Thermal Diffusivity, sq ft per sec
200	0.0075	0.118	1.30×10^{-4}
400	0.0071	0.128	1.13
600	0.0066	0.137	0.98
800	0.0061	0.150	0.83
1000	0.0055	0.170	0.66
1200	0.0049	0.200	0.50
1400	0.0043	0.223	0.39

DISCUSSION

MR. J. R. KATTUS.[1]—Was any study made of different thermocouple wire sizes?

MR. J. F. BURCHAM (*author*).—An experimental study of the performance of various thermocouple wire sizes was not made. The basis for selecting 28 gage wire is discussed in the paper. In general, the smallest wire size that will give a mechanically reliable installation for a particular set of test conditions is the best size.

MR. O. O. MILLER.[2]—Have you tried to roll thermocouple wires down to a ribbon in order to make better contact with the surface whose temperature is being measured? I have done this quite successfully using other types of thermocouples to measure the temperature of tin cans.

MR. BURCHAM.—We did not consider this approach. I suspect that using ribbon type thermocouple wire would result in an installation which would be too weak to provide sufficient mechanical reliability under the handling and test conditions we encounter in our laboratory.

MR. D. D. SELTZER.[3]—How many tests were run to produce the graphical information? Were there half a dozen tests for the curves at each point?

MR. BURCHAM.—The results of the tests are based on four installations of each type. The results are representative of the performance of all identical installations within the testing accuracy of ±10 F or ±1 per cent of the indicated temperature, whichever is greater.

MR. SELTZER.—Are you working toward a higher temperature than was indicated in the paper inasmuch as some of us are in ranges beyond 1600 and up to 3000 F?

MR. BURCHAM.—The maximum temperature in which we are primarily interested at the present time is about 1800 F, and at this temperature alumel-chromel thermocouple wire is suitable for use. Consequently, we have not experimented with other types of thermocouple wire. I think the suitability of a particular type of installation is dependent upon the heating rate; the temperature is relatively unimportant.

MR. F. W. DEMONEY.[4]—Have you looked into the use of thermocouple clamps in which you clamp each side of the material with one wire from the thermocouple pair? We have found this particularly effective in aluminum alloys.

MR. BURCHAM.—We have not found a clamping method to be satisfactory for measuring temperatures during radiant heating or transient temperature tests. The clamp components introduce heat sink and shielding effects. Design problems occur when trying to use clamping methods on large structures at points at which access paths are not present between opposite sides of the specimen. The method will not give the temperature of one surface but rather the average temperature of the two surfaces.

---

[1] Southern Research Inst., Metallurgy Division, Birmingham, Ala.

[2] Head Steel Section, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

[3] Chief, Quality Materials Laboratories, The Martin Co., Baltimore, Md.

[4] Head, Mechanical Metallurgy and Evaluation Branch, Kaiser Aluminum and Chemical Corp., Spokane, Wash.

MR. DEMONEY.—The depth of your thermocouples varied. Some were mounted on the surface, some were in drilled holes. If radiant energy was measured, you would probably have a gradient effect depending upon the material you used.

MR. BURCHAM.—The location of the thermocouple junction in relation to the point at which temperature measurements are required is very important. One purpose of this paper was to demonstrate the significance of this fact. The remoteness of the junction from the surface was one of the main reasons for the inaccuracies noted. Many thermocouple installations which have been used for steady state temperature measurements such as encountered in furnace heating cannot be used for transient measurements because gradients will exist between the thermocouple junction and the specimen. I hope this paper will discourage the use of just any type of thermocouple installation for rapid heating conditions unless it has been demonstrated that it will give accurate measurements.

MR. DEMONEY.—What is your standard of accuracy? What did you use as your control? The inaccuracy was so many degrees Fahrenheit. What did you use as a direct comparison?

MR. BURCHAM.—The methods of control and comparison and the accuracy are discussed in detail in the paper. In general, we accepted the temperatures measured on the unexposed surface of each panel as accurate and corrected for the calculated temperature gradient through the test panel. A good standard for measuring the temperature of surfaces heated by a radiant method does not exist for the temperature range investigated. Although some of the thermocouple installations may be more accurate than reported, those with inaccuracies of 40 F or greater are definitely unacceptable.

MR. R. W. FENN, JR.[5]—The author is to be commended for his excellent study of temperature measurement problems using the radiant-heating method. Others should be encouraged to investigate the temperature measurement problems accompanying rapid heating with the resistance and induction heating techniques. In one of our recent test programs[6] in which sheet-compression test specimens were rapidly heated by the resistance-heating technique, we had occasion to compare temperatures measured by thermocouples welded to the test specimen and thermocouples clamped to the test specimen. Comparison between (1) temperatures obtained with No. 30 gage iron-constantan thermocouples threaded through small pieces of ceramic insulator and clamped onto the specimen and (2) No. 30 gage iron-constantan thermocouples capacitance welded to the specimen showed no detectable difference (readability = 2 F) at 500 F. It should be noted, however, that the clamped thermocouples were inserted through holes in the specimen-supporting jig which also was heated quite rapidly by contact with the test specimen. Different results might be expected where specimens and thermocouples were exposed and permitted to radiate to the atmosphere. In a second comparison, compressometer gage points were made of $\frac{1}{8}$ in. diameter iron and constantan wires to permit temperature measurement by clamping the compressometer onto the specimen. Comparison of the temperatures measured with the compressometer to those obtained with No. 30 gage iron-constantan thermocouples welded to the specimens showed the apparent temperatures measured with the compressometer to be approximately 25 F lower than those obtained with thermocouples attached by welding. This large difference in temperature readings is attributed to the relatively large mass of the compressometer collars which prevents them from attaining the same temperature as the specimen in the short time required (~1 min).

---

[5] Chief of Testing and Instrumentation, Metallurgical Laboratory, The Dow Metal Products Co., Midland, Mich.

[6] R. W. Fenn, Jr., "Compression Testing of Sheet Magnesium Utilizing Rapid Heating," see p. 940.

MR. BURCHAM.—We have used some of the installation methods discussed in the paper for materials testing in which specimens were heated using resistance methods. In general, those methods which are suitable for radiant heating can also be used for resistance heating. Notable exceptions are those installations which introduce unacceptable discontinuities. The problem remains the same except that the source of the transient heating is the specimen. Those installations which exhibited erroneously high temperatures during radiant heating will register too low during resistance heating.

MR. W. J. DEAN.[7]—Has reflectivity anything to do with the inaccuracies obtained? In other words, did you make any attempt to determine whether or not the relative colors of the thermocouples exposed to radiant energy compared with the color or surface condition of the specimens had any specific correlatible effects?

MR. BURCHAM.—The thermocouples, except for the insulation, and the surface of the test panels were covered with a graphite paint type coating. Consequently, surface emissivity characteristics were the same for the thermocouple and the specimen.

MR. DEAN.—Have you done or are you proposing to do any work relative to the application of thermocouples to plastic materials? Or do you know anyone who is?

MR. BURCHAM.—No. It is not thought possible to measure accurately the temperature of the surface of a non-conducting material subjected to radiant heating by using thermocouples. The temperature of the material just below the surface can be accurately determined by using thermocouple probes.

---

[7] Lead Engineer, Structures, Temco Electronics and Missiles Co., Dallas, Tex.

APPLICATION OF INDUCTION HEATING TO SHORT-TIME ELEVATED-TEMPERATURE TENSION TESTING*

By Albert P. Levitt[1] and Anthony G. Martin[1]

Synopsis

Techniques and apparatus were investigated and developed for conducting short-time elevated-temperature tension tests utilizing high-frequency induction heating. By this method, metal specimens are heated rapidly to a predetermined temperature and then loaded in tension. Proportional temperature control equipment for the induction heater was developed for this testing technique. Tension test data were obtained at 600, 800, and 1000 F on 120 plain and welded titanium alloy specimens. Typical results obtained are given, and the limitations and possibilities of the technique are discussed.

The full and proper utilization of materials for rockets and guided missiles is dependent on knowledge of the properties of the materials at high temperatures. Since some missile components attain high temperatures in a very short time, simulation of these conditions is required to obtain the necessary property data. For this purpose tension specimens must be heated to the test temperature in a matter of seconds.

Methods of rapid heating applicable to tension testing include induction heating, radiant heating, resistance heating (by direct application of electrical power to the specimen), and immersion in hot molten substances. Resistance heating and immersion heating techniques have been used generally in the past.

Induction heating and radiant heating appear to offer the following advantages over other heating methods:

1. Prevention of overheating of the necked-down region of the loaded specimen as necking progresses.
2. Heating of the specimen without direct contact of the specimen with the heat source.
3. Elimination of specimen contamination by the heating medium.

To exploit the capabilities of induction heating, an investigation was conducted on its use for short-time elevated-temperature tension tests. Techniques and equipment evolved were employed in tests on titanium alloy specimens for a project conducted for the Army Ballistic Missile Agency.

Apparatus and Specimens

Test System:

Prior to initiating this investigation, a search of the literature on induction heating was made. Two of the pertinent texts are referenced in this paper (**1**,**2**).[2] Since the literature reviewed yielded no information on an automatic control system for induction heating, work was initiated by the authors on such a system. Since then, however, other systems have been developed by manufacturers of industrial controls (3,4). The assembled test system consists of a 20-kva output induction heater used in conjunction with a Tinius Olsen universal testing machine. By means of an especially designed electronic control circuit, the test specimen mounted on the tension tester is rapidly heated by the surrounding induction coil to a predetermined temperature. The test specimen may be loaded before or after heating, as desired, and tested over a range of strain rates. Figure 1 shows the test specimen mounted in the induction work coil. A detailed description of each element of this testing system follows.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 25–July 1, 1960.

[1] Chief and Electronic Engineer, respectively, High-Temperature Materials Branch, Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

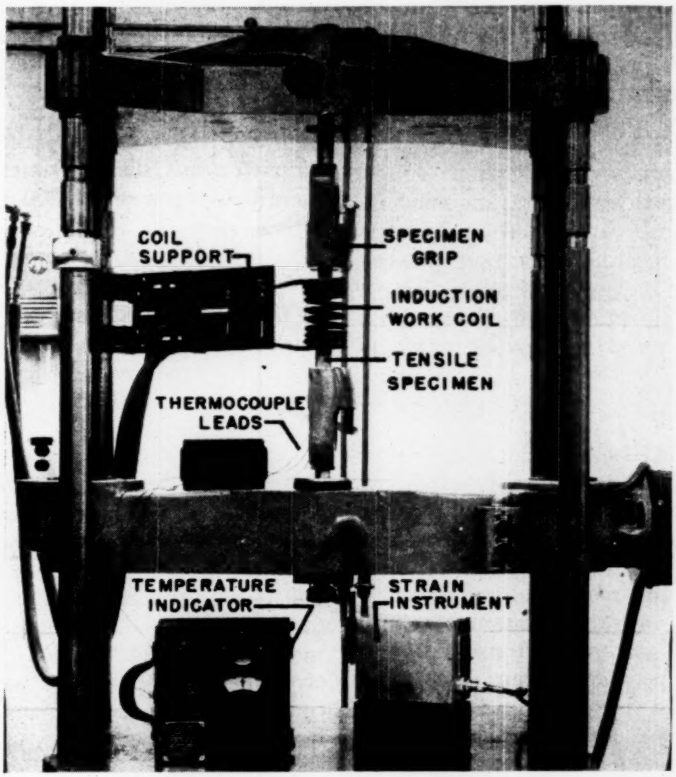


FIG. 1.—Test Specimen, Induction Coil, and Instrumentation.

Induction Heater:

The induction heater available for this study was a 20-kva output Scientific Electric unit operating at an output frequency of 450 kc. A three-phase full-wave rectifier provided the high d-c voltage that supplied the power for the oscillator circuit. Since the power output depends on the d-c voltage, it was decided to control temperature by controlling the d-c voltage. A control system for this purpose was developed by the authors and will be described later.

Induction Work Coil:

The induction work coil used consisted of nine turns of copper tubing, $\frac{5}{16}$ in. in outside diameter. The turns were spaced closely at the ends of the coil and farther apart at the middle. This compensated for greater heat loss at the ends of the specimens. The proper spacing was determined experimentally. The over-all size was approximately $5\frac{1}{2}$ in. in length and 3 in. in diameter.

Testing Machine:

A 60,000-lb capacity Tinius Olsen universal-type hydraulic testing machine equipped with an Olsen differential transformer-recorder was used in the tests. An Olsen universal strain instrument, in conjunction with the recorder, provided a record of crosshead travel *versus* load.

Test Specimen:

A total of 120 specimens, both welded and unwelded, were furnished by the welding research section of Watertown Arsenal Laboratories. The specimens were cut with their longitudinal axes parallel to the major rolling direction from sheets of various titanium alloys ranging in thickness from 0.031 to 0.078 in. Specimens from each alloy were divided into three groups: (1) unwelded base metal, (2) base metal welded at right angles to the major rolling direction and (3) base metal welded parallel to the major rolling direction. The weld beads were not machined flush. The over-all size of each specimen was 9 in. in length and 1 in. in width, and the gage section conformed to specifications for the sheet specimen of ASTM Method E 8.[3]

Temperature Sensing Elements:

Two types of high-temperature sensing elements are generally used for recording and controlling purposes—total radiation pyrometers and thermocouples. Either type can be used with the control apparatus that has been developed. Radiation pyrometers have the advantages of not requiring physical contact and of being usable at temperatures above the range of thermocouples. However, below 800 F the low voltage output of fast-response radiation pyrometers limits their usefulness; also, their accuracy depends on the thermal emissivity of the specimens. Consequently, for tests at 600, 800, and 1000 F, chromel-alumel thermocouples were used.

The thermocouple wire consisted of B&S gage No. 28 chromel and alumel wire with duplex insulation of fiber glass. The ends were bared, and the two wires were individually spot-welded on the specimens at points approximately $\frac{1}{32}$ in. apart. This method of joining the thermocouple to the specimen has the advantage of not interposing any mass between the thermocouple junction and the specimen's surface. Since the specimen and thermocouples are exposed to cool surroundings, a conventional thermocouple bead could introduce an error by having the actual point of contact between the two wires separated from the specimen's surface by the thermocouple bead.

Three such thermocouples were located at the top, middle, and bottom positions on the test section. The middle thermocouple was used for measurement and control purposes. The end thermocouples served for checking temperature distribution. Filter circuits were placed between the thermocouples and the temperature measuring instruments to bypass induced radio frequency currents.

Temperature Indication:

The end thermocouples were connected to a twin-input hand-operated temperature indicator. The middle thermocouple was connected through a reference junction maintained at 150 F to a recorder-controller calibrated in millivolts and equipped with a control slide-wire. The slide-wire is mechanically linked to the recorder-pen drive such that the position of the wiper arm varies with the deviation of the temperature from the manually set control point, within a band width of ± 5 per cent of scale range.

---

[3] ASTM Methods of Tension Testing of Metallic Materials (E 8 – 57 T); 1958 Book of ASTM Standards, Part 3.

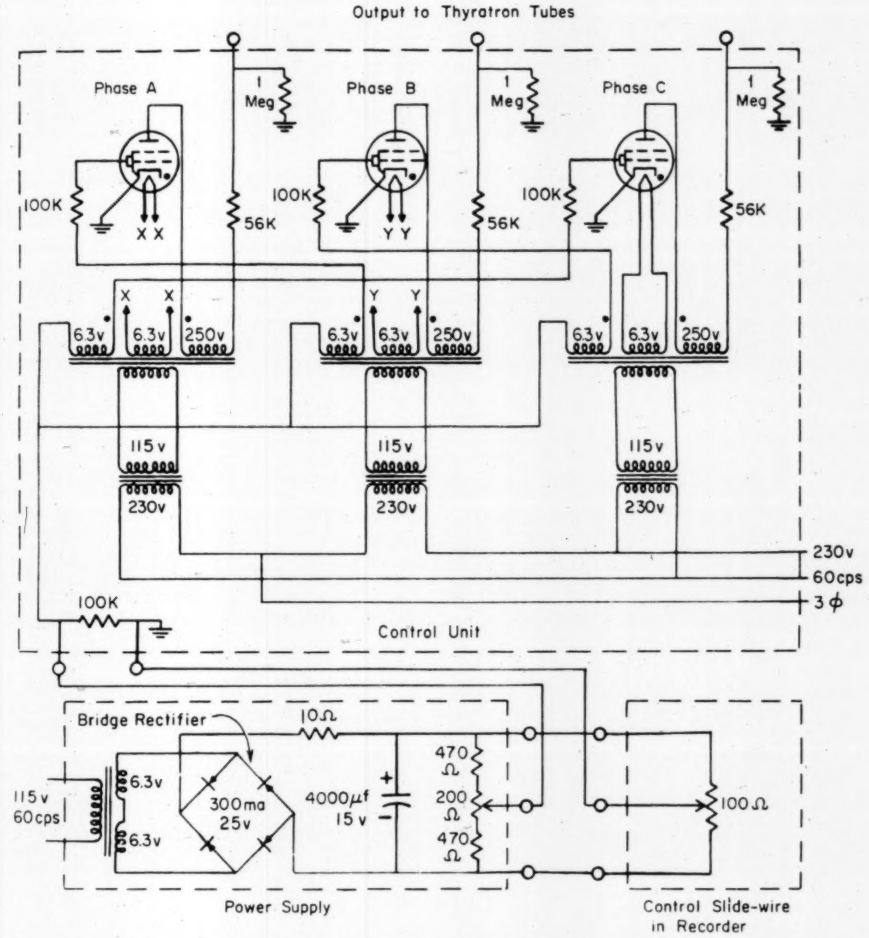

Output to Thyratron Tubes
Phase A
Phase B
Phase C
1 Meg
1 Meg
1 Meg
100K
100K
100K
56K
56K
56K
X X
Y Y
X
X
Y
Y
6.3v
6.3v
250v
6.3v
6.3v
250v
6.3v
6.3v
250v
115v
115v
115v
230v
230v
230v
230v
60cps
3 ϕ
100K
Control Unit
Bridge Rectifier
10Ω
300ma
25v
6.3v
6.3v
115v
60cps
4000μf
15 v
+
−
470 Ω
200 Ω
470 Ω
100 Ω
Power Supply
Control Slide-wire in Recorder

FIG. 2.—Circuit for Power Supply, Control Slide-Wire, and Control Unit.

Control Equipment:

Bridge Circuit.—The control slide-wire is connected into a bridge circuit powered by a 12-v d-c power supply. The bridge circuit and power supply are shown in Fig. 2. The bridge output is a d-c voltage that varies in amplitude and polarity with the temperature deviation. An adjustment is provided for varying the balance point slightly.

Control Unit. –The control unit converts variations of the bridge output into variations of the time at which control pulses are generated by miniature thyratrons. The circuit is illustrated in Fig. 2. For simplicity, approximations of thyratron characteristics are used in the discussion. It shall be assumed that (1) the tubes start conducting current at the instant the grid becomes positive, provided the plate is positive, and (2) once current flows, it will continue until the plate voltage approaches zero.

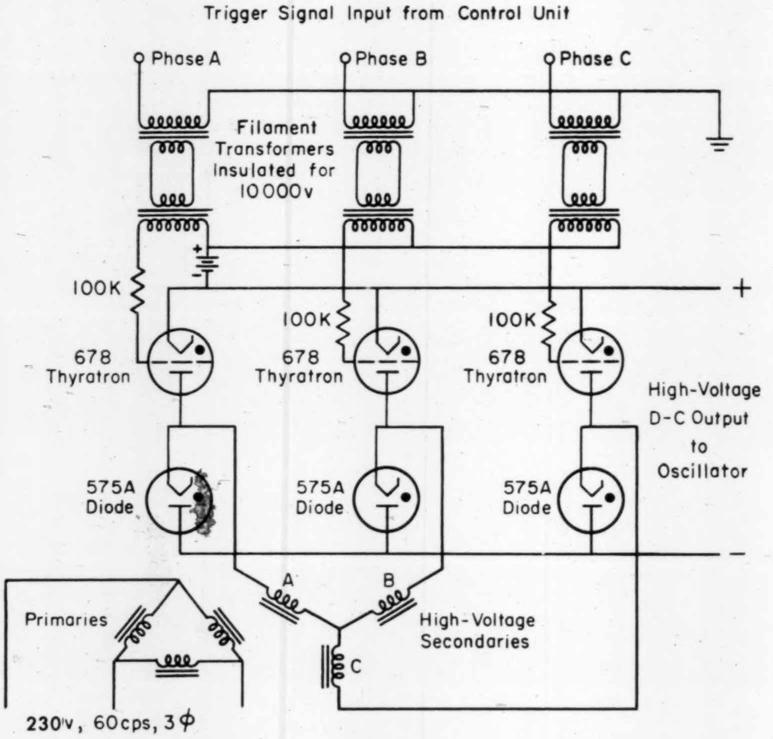

Trigger Signal Input from Control Unit
Phase A
Phase B
Phase C
Filament Transformers Insulated for 10000v
+
-
100K
100K
100K
678 Thyratron
678 Thyratron
678 Thyratron
+
High-Voltage D-C Output to Oscillator
575A Diode
575A Diode
575A Diode
-
A
B
C
Primaries
High-Voltage Secondaries
230v, 60cps, 3φ

FIG. 3.—Circuit for Induction Heater Power Supply.

Voltage from phase A of the line voltage is applied to the plate of a 2D21 thyratron. Voltage from phase B, lagging phase A by 120 deg, is mixed with a variable d-c voltage and applied to the grid. The time at which the tube starts conducting is advanced or retarded by the variable d-c voltage.

When the tube fires, current instantly flows in the plate circuit and a pulse appears at the output transformer. A pulse is produced once every cycle. Its timing is controlled by the deviation of the temperature from the control point. The other phases produce pulses in the same fashion once every cycle, the pulses in any phase lagging or leading pulses in other phases by 120 deg. These pulses are applied to rectifier tubes in the induction unit rectifier section.

The rectifier section employed six type 575A diodes. Three of these were replaced by type 678 thyratrons (grid-controlled rectifiers) of equivalent current and voltage ratings, one for each phase. These thyratrons were placed in the positive side of the rectifier circuit to provide a common cathode connection.

A battery supply was added to bias the grids negatively, preventing current flow in the absence of control pulses. A pair of filament transformers with high-voltage insulation were connected back to back to couple each grid to the proper output signal from the control unit. This circuit is illustrated in Fig. 3.

Power control is achieved by controlling, in each cycle, the time during which the rectifier tubes conduct. When a control pulse appears in the grid circuit of one phase, it overcomes the negative bias voltage and permits current flow which then continues until the plate-to-cathode voltage approaches zero.

In a three-phase full-wave rectifier, the voltage across each thyratron is not a simple *sine*-wave voltage; it is a double-peaked voltage due to added components from the other phases. Maximum output is obtained with the trigger pulse at 30 deg. When the pulse is retarded in response to a rise in temperature (as the temperature approaches the control point), the output drops. If the temperature keeps rising, the output gradually drops until it reaches a low level (as the control pulse approaches 180 deg) and then abruptly drops to zero. Unless the input required is very low, a condition of equilibrium is rapidly reached, such that the input power is just sufficient to maintain the temperature of the specimen at a level within the control band.

OPERATING PROCEDURE

Very fine scribe marks were made at each end and at the middle of the 2-in. test section of the specimen. Thermocouple wires were then attached at each mark, using a Unitek spot-welder. The thermocouple wires were then connected through radio frequency filter networks to the temperature indicating and controlling instruments. The specimen was placed inside the induction coil and inserted in V-grips to a depth of 1 in. at each end. The control indicator was set manually to a point slightly less than that corresponding to the desired temperature.

The induction heater power was then turned on. After the temperature had risen and became stabilized, a readjustment was made in the control indicator to bring the temperature to the desired point. The temperatures at the ends of the test section were checked. If the over-all temperature spread was more than 20 F, the power was turned off and the thermocouples and the location of the specimen within the coil were checked. Any required adjustments were then made. With a few exceptions, the temperature difference was kept well below 20 F.

The exceptions were among the welded specimens, particularly the specimens welded transverse to the major rolling direction. These developed a higher temperature at the middle (the beaded weld area) than at the ends due to the greater thickness of the weld area. For this reason, in these specimens the middle thermocouple was placed just above the weld zone. This procedure was adopted after observing in initial tests that the transversely welded specimens all fractured in the base metal and produced strength-temperature curves almost identical with those for the base metal. The longitudinal welds did not present this problem since the welds ran along the length of the specimens.

DISCUSSION

The heating rate of the specimens was observed to vary considerably with variations in thickness. It may be noted that the heating rate decreased with reduction in thickness. The reason for the decrease in heating rate can be understood by considering the effects on heat input of thickness, electrical and magnetic properties, and induction heater frequency. Figure 4 shows curves plotted from experimental data for average total time required to reach and level off at each test temperature for each thickness.

A characteristic of induction heating is that maximum heating takes place at the surface of electrically conductive materials. This is due to the fact that the current density decreases exponentially with increasing depth from the surface. The depth at which the current density is 36.8 per cent of the surface value is known as the depth of penetration and is given by the equation:

$$\delta, \text{cm} = \frac{1}{2\pi}\sqrt{\frac{\rho}{\mu f}}$$

or

$$\delta, \text{in.} = 0.0627\sqrt{\frac{\rho}{\mu f}}$$

where:

δ = depth of penetration,

ρ = resistivity in microhm-cm,

μ = relative magnetic permeability, and

f = frequency in kilocycles per sec.

For a frequency of 450 kc and for nonmagnetic materials or magnetic materials at temperatures above the Curie point, where magnetic properties disappear ($\mu = 1$), the equation above for the depth of penetration may be expressed as follows:

$$\delta, \text{in.} = 0.00295\sqrt{\rho}$$

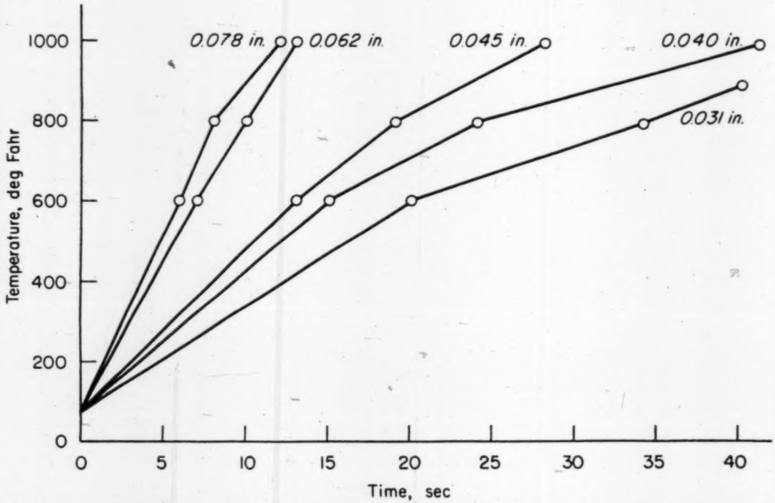


FIG. 4.—Average Time to Heat Titanium Specimens of Different Thickness to Equilibrium Temperatures.

For magnetic materials below the Curie point, δ is much smaller, since μ is very large.

The average power density in an inductively heated strip (whose thickness is many times smaller than the width) is related to the ratio of thickness to depth of penetration by the equation (5):

$$p/v = \tfrac{1}{4}\, Ho^2 \mu f \frac{\delta}{t}\left(\frac{\sinh t/\delta - \sin t/\delta}{\cosh t/\delta + \cos t/\delta}\right)$$

where:

p/v = power density in watts per cu cm,

Ho = peak magnetizing force in oersteds,

μ = relative magnetic permeability,
f = frequency in cycles per sec,
δ = depth of penetration, in., and
t = specimen thickness, in.

If thickness is the only variable in the right-hand side of the equation, the power density becomes:

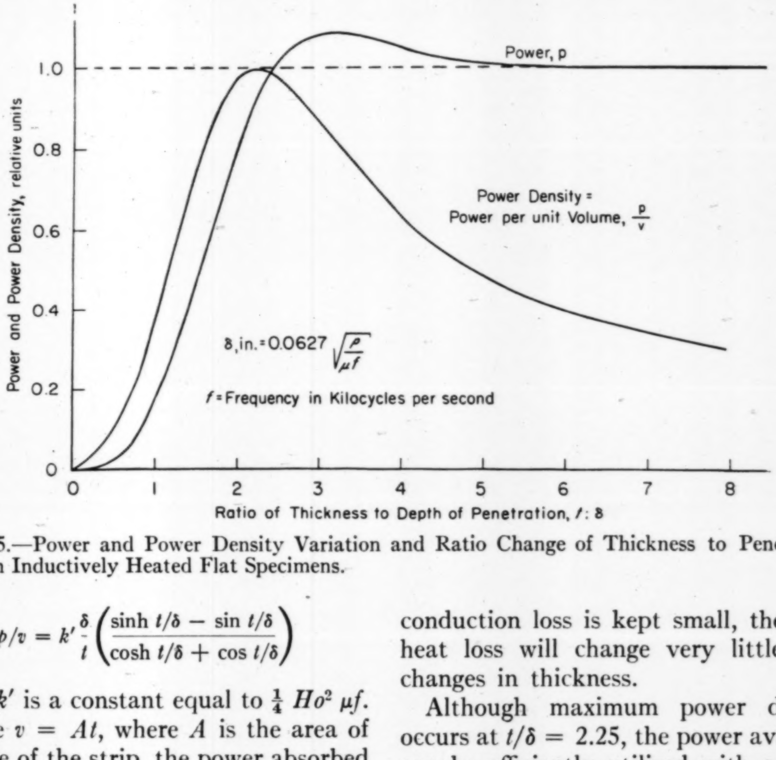


FIG. 5.—Power and Power Density Variation and Ratio Change of Thickness to Penetration Depth in Inductively Heated Flat Specimens.

$$p/v = k' \frac{\delta}{t} \left(\frac{\sinh t/\delta - \sin t/\delta}{\cosh t/\delta + \cos t/\delta} \right)$$

where k' is a constant equal to $\frac{1}{4}\, Ho^2\, \mu f$.

Since $v = At$, where A is the area of one side of the strip, the power absorbed is:

$$p = k'' \left(\frac{\sinh t/\delta - \sin t/\delta}{\cosh t/\delta + \cos t/\delta} \right)$$

where k'' is a constant equal to $Ak'\delta$.

Power density p/v and power p are shown as functions of t/δ in Fig. 5. The power density rises rapidly from zero thickness to maximum or near maximum at values of t/δ between 2.0 and 2.5 and then diminishes gradually with increasing thickness. The absorbed power rises rapidly to a maximum value at $t/\delta = \pi = 3.14$ and then gradually settles to a slightly lower level.

For the thicknesses involved, the total surface area can be considered to vary only slightly. Heat loss through convection and radiation are then practically independent of thickness. If conduction loss is kept small, the total heat loss will change very little with changes in thickness.

Although maximum power density occurs at $t/\delta = 2.25$, the power available can be efficiently utilized with a much greater thickness. However, the time required to raise the greater mass to a given temperature is correspondingly increased.

As t/δ is reduced below 2, both power density and absorbed power decrease rapidly. Not only is the heating rate reduced, but the maximum attainable temperature is less.

The resistivity of pure titanium increases from 55 microhm-cm at 68 F to 142 microhm-cm at 1000 F. Since pure titanium is not magnetic ($\mu = 1$), the depth of penetration at 450 kc changes from 0.022 in. at 68 F to 0.035 in. at 1000 F. The optimum thickness correspondingly changes from 0.050 in. at 68 F to 0.079 in. at 1000 F. The thicknesses of the specimens tested ranged from 0.031 to 0.078 in. Their resistivities were not determined. However, the heating rates obtained are consistent with resistivities of approximately the same magnitude as for pure titanium. For thicknesses of less than 0.062 in. the rate of heating decreases rapidly as the thickness is reduced.

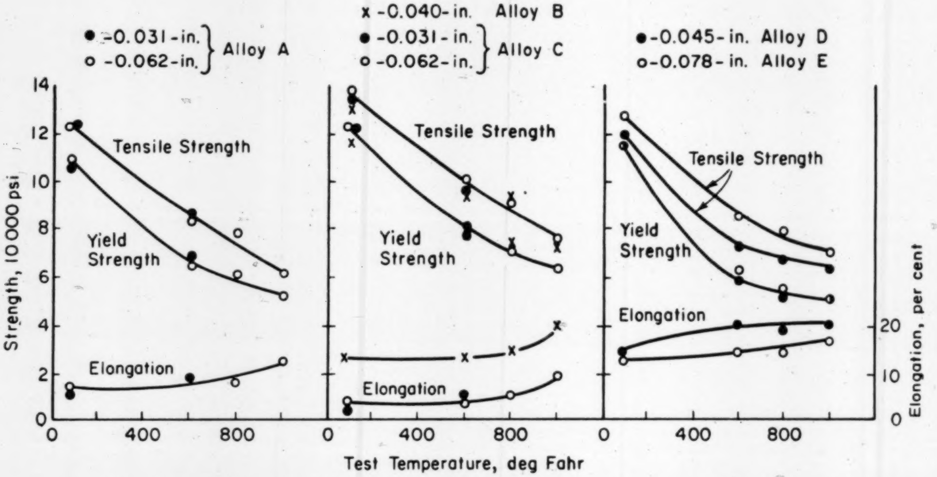


FIG. 6.—Elevated-Temperature Tensile Properties of Titanium Alloys as Obtained by Short-Time Induction-Heating Techniques (6).

It did not prove feasible to heat the thinnest specimens to 1000 F because of overheating of the specimen-gripping apparatus. The situation can be improved by changing the over-all length of the specimens from 9 in. to 12 in. This would permit greater separation between the induction coil and the grips and minimize induced currents in the grips.

With few exceptions, the time taken to heat each specimen to the test temperature and to make necessary adjustment ranged from 20 to 60 sec. An additional 10 to 30 sec were expended in testing the specimen at a platen speed of approximately 1 in. per min. Temperatures at the middle were maintained within ± 10 F of the desired temperatures throughout the test. In 27 cases out of 120, the specimens required readjustment due to excessive thermal gradients. The additional time during which the specimens were heated did not cause any discernible change in the mechanical properties.

The tests were made in duplicate at 600, 800, and 1000 F. Data showing yield strength, tensile strength, and elongation of the base metal as a function of temperature are plotted in Fig. 6. An earlier report (6) containing full details on the mechanical test results points out that the strength-temperature curves of the specimens welded transverse to rolling were almost identical with those for the base metals and that the specimens composed of base metal welded parallel to rolling possessed greater strength.

The determinations of yield strength

and ultimate strength were made from recordings of crosshead travel *versus* load. Although absolute values of yield strength cannot be obtained by measurement of crosshead motion, this method does provide useful comparative data. The results obtained showed good reproducibility. However, the slope of a given curve in the elastic region is affected by elastic deformation of portions of the specimen beyond the gage length, the specimen holders, and the testing machine. Thus, Young's modulus cannot be determined from these curves without applying proper corrections.

A conventional extensometer was not used to measure strain because of the susceptibility of the metal parts to overheating in the induction field. An improved extensometer under development at Watertown Arsenal Laboratories consists of components made of ceramic materials that are unaffected by the induction field.

The rates of heating and the test data obtained met the specific requirements. The rate of heating can be raised further by decreasing the heat losses. Since power input is dependent on the coupling between the coil and the specimen, it can be increased by reducing the coil diameter, thus reducing the distance between the coil and the specimen. However, the diameter should not be reduced to the extent that localized zones of heating occur in the areas of the specimen closest to the coil turns.

Along with increasing the efficiency of power transfer, it is important to reduce heat losses. Causes of heat loss, in order of importance for this application, are: radiation, convection, and conduction. Radiation can be reduced by a reflective heat shield between the coil and the specimen. Convection can be controlled by forming a closed chamber around the specimen which may be evacuated if desired. Conduction losses are small since the cross-section areas of the specimens are small. They can be reduced further by increasing the length of the specimens as was suggested for preventing the overheating of specimen grips. With proper control of these factors, rates of heating of several hundred degrees per second should be possible with the present equipment.

While the highest test temperature reported here is 1000 F, this equipment is capable of testing specimens at temperatures higher than 2500 F. It is anticipated that temperatures, heating rates, and strain rates to be encountered in future missile systems will continue to increase. Therefore, effort is currently directed at greatly extending the capacity of this system by appropriate modification or replacement of the units of the present system.

SUMMARY AND CONCLUSIONS

Induction heater control apparatus was developed and techniques evolved for the use of induction heating in short-time high-temperature tension tests. The apparatus and techniques were used successfully in conducting tension tests on titanium specimens at temperatures of 600, 800, and 1000 F. With few exceptions, the total time expended for heating and testing a single specimen was less than $1\frac{1}{2}$ min of which the major portion was heating time. It appears possible to make further substantial increases in the capacity of the system, and work is in progress to accomplish this.

Based on the foregoing results, it may be concluded that induction heating is well suited to heating specimens for short-time high-temperature tension tests. It has the following advantages:

1. It prevents overheating of the necked-down region of the loaded specimen as necking progresses.
2. It permits heating the specimen without direct contact of the specimen with the heat source.
3. It eliminates specimen contamination by the heating medium.

REFERENCES

(1) F. W. Curtis, "High Frequency Induction Heating," McGraw-Hill Book Co., Inc., New York, N. Y., 2nd Edition (1950).

(2) J. Wesley Cable, "Induction and Dielectric Heating," Reinhold Publishing Corp., New York, N. Y. (1954).

(3) J. V. Metzger and W. W. O'Hara, "New Controls for Crystal Growing," *Instrumentation*, Minneapolis-Honeywell Regulator Co., Vol. 9, No. 5, p. 17 (1956).

(4) E. T. Davis, W. B. Alden, and F. H. Wyeth, "Precise Heat for Growing Silicon Crystals," *Electronics*, Vol. 30, No. 2, p. 164 (1957).

(5) Industrial Electronics Reference Book, Westinghouse Electric Corp., p. 381 (1948).

(6) C. E. Hartbower and G. M. Orner, "Feasibility of Titanium for Welded Missiles," *Technical Report*, WAL TR 401/301, Watertown Arsenal Laboratories, Watertown, Mass., Aug. 1958.

DISCUSSION

MR. S. E. BRAMER.[1]—When the author and others talk about heating systems they usually bring up the old bugaboo that resistance heating gives erroneous results because of necking of the specimen and overheating. I think we all forget some basic fundamental physical phenomena. We do not have any necking of the specimen up to the proportional limit. From the proportional limit to ultimate strength we get uniform necking; with adequate equipment there is such excellent control that uniform necking does not affect the temperature control and there is no temperature rise. However, there is local necking at the maximum load which corresponds to the material's ultimate tensile strength. This is where erroneous results will be obtained, and the only thing affected is total elongation. Besides, total elongation is not affected very much because the tests are carried out so rapidly that failure occurs in a very short time. In design, people are not interested in what happens beyond the ultimate strength. They are only interested in ultimate tensile strength, and as long as you can pick that up before local necking takes place, we need not worry about the differences between resistance heating, induction heating, immersion heating, and radiant heating. They are all good methods for testing at elevated temperatures. However, I do not know of any method that allows one to make as many tests in as short a time as resistance heating with its ease of setup and its ease of attaching an extensometer.

MR. A. P. LEVITT (*author*).—I do not wish to belabor the point between the advantages of induction heating versus resistance heating. The primary objective of our paper was to point out that induction heating can be used to advantage for short-time high-temperature tension tests. As you mentioned, all of these techniques have their advantages, and whether we use one or the other depends upon the circumstances. In our particular instance, the test data which we obtained met our requirements and were very satisfactory, and we have adapted induction heating to a number of other tests also. For example, we have adapted it for conducting measurements of the elastic constants of metals and alloys at elevated temperatures; in this particular case the induction heating is so useful that we can do this in a matter of minutes once the apparatus is set up. I do not wish to disparage resistance heating. I simply say that induction heating is useful also.

---

[1] Head, Fabrication Development Section, Hughes Tool Co., Aircraft Division, Culver City, Calif.

It should also be noted that there are numerous investigators who are concerned with what happens to the material beyond its ultimate strength, because this behavior sheds more light on the basic and extremely important processes of deformation and fracture of materials at elevated temperatures.

THE PHENOMENON OF STRESS-RELAXATION*†

By A. M. Freudenthal[1]

Synopsis

The phenomenon of stress-relaxation is discussed in relation to its physical origin, its mathematical representation in one as well as in three dimensions, and its experimental determination, with particular emphasis on the interrelation between creep and relaxation. It is shown that neither the direct relaxation test nor the conventional conversion of creep into relaxation data provide reliable and directly useful design information concerning the relaxation behavior of the material.

The decrease with time of the intensity of the stress induced in a solid or pseudosolid medium by an initially imposed and sustained constant strain is known as "stress relaxation." Phenomenologically, the process is the result of the conversion of the initial elastic strain into irrecoverable strain of the same magnitude, through inelastic deformation of the medium proceeding under the decreasing stress. Structurally, the process represents the gradual approach of the mechanically and thermodynamically unstable, elastically distorted crystal aggregate or molecular arrangement towards a condition of thermodynamic equilibrium.

The mechanisms by which the stress-relaxation is accomplished are of various origins; they may involve the motion of individual particles (atoms or molecules), of limited groups of particles (molecular chains), or of elements of microscopic size (crystallites and crystals). Apart from the medium itself, the process is necessarily affected by the degree of thermodynamic disequilibrium between the medium and its surroundings, since the re-establishment of thermodynamic equilibrium is itself a relaxation process. The associated "diffusion" processes (heat flux, magnetic flux, or particle flux) may be confined within the solid itself or may represent exchanges with the surrounding medium.

The practical importance of the phenomenon of stress-relaxation is in the fact that where such relaxation is present it determines the intensity of the stresses induced by imposed deformation ("deformation stresses"); the stresses resulting directly from the applied forces ("load stresses") are not affected, unless their intensity and distribution depends significantly on the deformation of the structure, as in all cases when the load stresses cannot be obtained from the stress-equilibrium conditions alone. Sig-

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26-July 1, 1960.

† This research was supported by the U. S. Air Force under contracts AF 33(616)-2729 and AF 33(616)-6112, monitored by the Materials Laboratory, Wright Air Development Division, Wright Patterson Air Force Base, Ohio.

[1] Columbia University, New York, N. Y.

nificant stress-relaxation is present even when the total amount of irrecoverable strain, ϵ'', is very small, since it depends essentially on the product of this strain and the elastic modulus E according to the approximate uniaxial relation $d\sigma/dt = \dot{\sigma} = -Ed\epsilon''/dt = -E\dot{\epsilon}''$, rather than on ϵ'' alone; thus it may affect the state of stress considerably even when the creep deformation itself, because of the very low rate at which it proceeds, is not a significant design parameter.

The reliable evaluation of the relaxation behavior of a material by experiment is rather difficult because of the difficulty of applying initial strain rapidly enough to ensure purely elastic response, and subsequently to control its value accurately enough during the test. Because of these difficulties and because of the relative simplicity of creep-testing equipment it has been a widespread practice to determine the relaxation characteristics of a material from creep tests, on the basis of the assumption that the inelastic strain, ϵ'', that is produced during a relaxation test proceeds by the same mechanism as in a creep test. By neglecting the effect of creep recovery and by introducing the irrecoverable creep rate in either of the alternative forms, $\dot{\epsilon}''(\sigma,t)$ ("time-hardening"), or, $\dot{\epsilon}''(\sigma, \epsilon'')$ ("strain hardening"), or their combination, $\dot{\epsilon}''(\sigma, \epsilon'', t)$ into the relation $\dot{\sigma} = -E\dot{\epsilon}''$, the relaxation function, $\sigma(t)$, can presumably be obtained by integration, provided the (initial) stress, σ_0, at the start of the relaxation process, $t = 0$, can be expressed as a simple function of the initially applied strain $\epsilon(0) = \epsilon_0$ of the form $\sigma_0 = E\epsilon_0$ or $\sigma_0 = E'\epsilon_0$. The above assumption implies a specific dependence of the momentary inelastic strain rate (and thus of the momentary rate of stress-relaxation) on the momentary stress rate, while the provision implies that the time-independent initial stress is either purely elastic, with a constant elastic modulus, E, or elastic-plastic with secant modulus E'.

For instance, the consideration of a set of creep functions obtained from experiments under constant stress of the frequently used form $\epsilon'' = f(\sigma)t^n$ (Nutting equation) (**1**),[2] as a general stress-strain-time relation valid for variable stress, implies a linear stress-rate effect valid for $\sigma \gtrless 0$ of the alternative forms:

$$\frac{d}{dt}\ln \epsilon'' = n/t + \frac{f'(\sigma)}{f(\sigma)}\,\dot{\sigma} \quad \text{or} \quad n\left[\frac{f(\sigma)}{\epsilon''}\right]^{1/n} + \frac{f'(\sigma)}{f(\sigma)}\,\dot{\sigma} \ldots (1)$$

For a specified total creep strain, ϵ'', therefore

$$\frac{d\sigma}{dt} = -\frac{nf(\sigma)}{tf'(\sigma)} \quad \text{or} \quad n\,\frac{[f(\sigma)]^{(n+1)/n}}{f'(\sigma)} \cdot \text{const} \ldots (2)$$

Specifically, for time-hardening creep with $f(\sigma) = ae^{b\sigma}$ and $f'(\sigma) = ab\,e^{b\sigma}$, or $f(\sigma) = c\sigma^m$ and $f'(\sigma) = mc\sigma^{m-1}$.

$$\frac{d\sigma}{d\ln t} = -\frac{n}{b}, \quad \text{or} \quad \frac{d\ln\sigma}{d\ln t} = -\frac{n}{m}$$

Thus in logarithmic time scale the $(\sigma - \ln t)$ or $(\ln\sigma - \ln t)$ diagrams for different values of the parameter ϵ'' (creep-strain) are families of parallel straight lines with slope $(-n/b)$ or $(-n/m)$ respectively.

Introducing an arbitrarily selected number of time intervals, θ_1, into the Nutting equation the resulting equations $\epsilon'' = f(\sigma)\theta_1^n$ represent a family of fictitious relations between ϵ'' and σ associated with constant times, so-called "isochronous stress-strain relations." In reality, however, these relations are not differentiable and the apparent "tangent moduli"

$$(\partial\sigma/\partial\epsilon'')_{\theta_i} = E''_{\theta_i} = \theta_i^{-n}/f'(\sigma) \ldots (3)$$

physically inadmissible, since the constant stress tests from which they are derived do not a priori justify extrapolation to conditions of variable stress. Attempts to produce relaxation diagrams, $\dot{\sigma} < 0$, from isochronous stress-strain diagrams (2) are therefore of doubtful validity.

---

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

Various degrees of agreement between the results of acutal relaxation tests and those predicted on the basis of creep tests have been reported, and various procedures have been proposed for the conversion of creep test results into relaxation data (3), based on the assumptions made above. However, only for monomeric materials with relatively simple structures can good agreement be expected, provided the creep recovery (after-effect) is of minor significance; however in metals as well as in many high polymers, the mechanisms of inelastic deformation are different under conditions of creep and of relaxation.

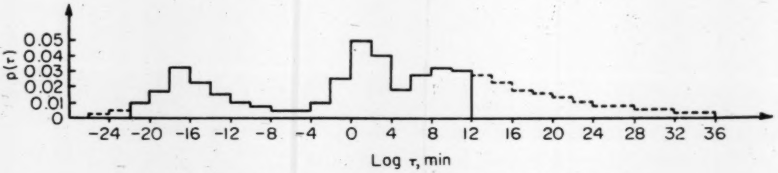


FIG. 1.—Relaxation Spectrum of Hard Phenolic (5).

PHYSICAL ORIGIN OF STRESS-RELAXATION

Atomic and Molecular Processes:

Significant (shear) stress relaxation in metals due to migration of individual solute particles arises when such motion affects the resistance to slip along potential slip planes or to quasi-viscous flow along grain boundaries. An imposed nonhomogeneous field of elastic strain will induce a preferential equilibrium distribution of solute interstitial atoms and lattice imperfections, associated with a criterion of minimum elastic energy of local distortion of the lattice. It induces therefore diffusion of solute particles and lattice imperfections from the stressed to the unstressed equilibrium positions, changing the resistance to flow or slip along planes or around regions from which or into which the imperfections migrate, and unblocking or blocking the motion as well as the resulting relaxation of shear stresses within the potential planes or regions of motion (4).

Stress-relaxation in high polymers is essentially due to changes of configuration of long-chain molecules by thermal motion (translation and rotation), as a result of which potential energy stored in the chain molecules is gradually dissipated. In polymers of high molecular weight, the extent of motion of molecular chains shows a very wide range of variation from short links to complete chains; a wide range of relaxation times is therefore a characteristic of such materials. Their relaxation behavior can be described by a "spectrum" of relaxation times representing the specific contributions of the individual configurational changes to the total relaxation. Figure 1 shows the wide relaxation spectrum of a hard cotton-filled high molecular weight phenolic (5).

Alternative mechanisms of stress-relaxation in cross-linked polymers, particularly in the rubberlike state, involve dissociation of molecular bonds by mechanical breakdown, by chemical reaction ("scission"), and by crystallization.

Quasi-Viscous Relaxation Centers:

Shear-stress relaxation can be associated with the existence of microscopic or macroscopic localized regions of essentially viscous response within a continuous elastic matrix; such regions can be considered as "centers of relaxation" (6, p. 126). Their quasi-viscous response is due to the relatively high degree of disorder in their atomic or molecular structure. Since there is an ordering tendency in regions of localized structural disorder of crystalline substances, their quasi-viscous character may be transient, provided the rate of self-diffusion is sufficiently high to make the results of this tendency significant within the experimental time scale.

Relaxation centers in metals are formed by grain boundaries and by slipbands; in polymers they are made up of the regions of low polymerization. The quasi-viscous nature of the grain boundaries has been confirmed by experiments of many investigators (6, p. 150) and with it that of the mechanism of viscoelastic shear-stress relaxation within the grain boundaries of polycrystalline metal aggregates. Similarly, the quasi-viscous nature of newly formed slipbands due to the disordered structure produced within the slipped region has been confirmed by experiments (6, p. 136) and shown to be consistent with the concept of high concentrations of dislocations induced by the deformation. The only difference in the nature of the viscous response between grain boundaries and slipbands is due to the lower thermal stability of the slipband structure, which favors its rapid readjustment and absorption into the surrounding crystal matrix, with consequent loss of viscous character, particularly at elevated temperatures (7).

The rapid relaxation of shear stresses along the newly formed, quasi-viscous slip regions in the course of the deformation provides an important mechanism for the spreading of slip, since under constant or increasing over-all stress new potential slip planes are continuously called upon to carry the local excess stresses transferred from the previously formed slip planes on which the shear-stress relaxation has been essentially completed. Under decreasing stress, however, the permanent deformation by the generation of new slipbands is blocked if the rate of decrease of over-all stress is higher than the rate of shear-stress relaxation along the previously formed slipbands. Such conditions may easily arise in relaxation tests, during the early stages of rapid decrease of stress.

While under conditions of loading $\dot{\sigma} > 0$, shear-stress relaxation within slipbands and grain boundaries in polycrystalline aggregates raises the stresses in the crystalline regions, and local shear-stress relaxation in low-polymerized regions of crystalline polymers causes the transfer of stresses into the crystalline domains, the existence of such relaxation centers gives rise to gradual recovery, upon or during unloading, of that part of the deformation of the crystalline (elastic) domains or of the region of comparatively high viscosity, that was caused during loading by the stress-relaxation within the operating relaxation centers ("strain recovery" or delayed elastic deformation). If in the straining of a polycrystal new permanent relaxation centers are produced as a result of crystal fragmentation following extensive slip and grain dissociation (8), the extent of strain recovery following unloading will increase, making it appear as if part of the slip deformation could actually be recovered, while, in fact, it is only the additional delayed elastic deformation associated with the shear-stress relaxation in the newly formed centers that is recovered by their (viscous) interaction with the surrounding elastic matrix. Because of this multiplication of potential relaxation centers in the course of plastic deformation, the extent of strain recovery increases with the amount of preceding plastic strain.

Strain recovery is also affected by temperature since it depends on the apparent viscosity of the operating relaxation centers, which is strongly temperature-sensitive. Thus, while in a polycrystalline metal at low temperatures only the highly distorted slip planes may be effective as (transient) relaxation centers, stress-relaxation upon loading and strain recovery following unloading will become the more significant the higher the temperature and the more extensive therefore the grain-boundary relaxation (**9**), provided the temperature is not high enough to produce extensive "flow" by grain-boundary rotation and dissociation.

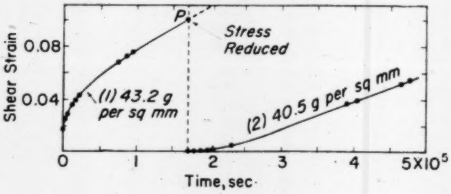


FIG. 2.—Effect of Stress-Reduction on Strain Rate in Zinc Crystals (**11**).

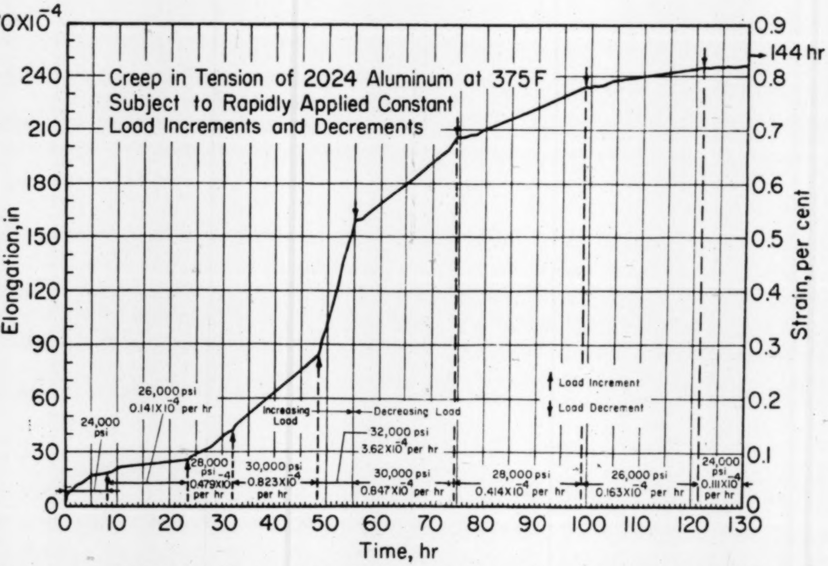


FIG. 3.—Creep of 2024 Aluminum at 375 F Under Constant Stress Increments and Decrements of 2000 psi.

Quasi-Homogeneous Viscoelasticity and Plasticity:

In metals at elevated temperatures nonlinear flow (creep) is due to the interaction of quasi-viscous grain-boundary slip and rotation with transcrystalline slip and grain dissociation; stress relaxation can be ascribed to the existence of quasi-homogeneously distributed relaxation centers. A significant effect of the stress rate on the nonlinearity of the relation between strain rate and stress arises from the mechanism of slip: to produce appreciable deformation within a crystal structure some multiplication of dislocations must occur, and the dislocations must be driven through the imperfect lattice against the resistance of solute particles and grain boundaries. Both processes encounter increasing resistance due to the gradual exhaustion of easily moveable dislocations and other imperfections; they will therefore be more or less rapidly blocked unless the applied stress is increased or, at least, kept constant. Appreciable transcrystalline slip can thus be expected only as long as $\dot{\sigma} > 0$.

Experiments with aluminum have shown that in fine-grained pure metal that fraction of total creep that is due to clusters of slipbands (coarse slip) is not less than about 0.20 of the total strain, increasing, for a coarse-grained aggregate, to almost 0.50 (10). The remaining deformation is the result of fine slip and quasi-viscous grain-boundary motion. Only this latter can be assumed to be largely independent of stress rate as the fine slip and grain-boundary motions are probably much less dependent on the existence of a value of $\dot{\sigma} > 0$ than coarse slip, which should be rapidly reduced or blocked when $\dot{\sigma} < 0$, the more sharply the more rapid the decrease of the stress.

Room-temperature creep experiments on single zinc crystals (11) confirm the effect of decreasing stress in blocking slip. Figure 2 shows the momentary blocking of the rate of creep produced by a sudden small reduction of the applied stress. Similar results have been obtained in experiments with lead and copper wire at room and slightly elevated temperatures (12).

In order to show that this phenomenon exists in polycrystalline aluminum at elevated temperatures, a creep test under stepwise increasing and decreasing stress was performed at Columbia University on standard creep specimens of 2024 aluminum at 375 F (Fig. 3). The complete blocking of creep for several hours following a sudden decrease of the stress by 2000 psi, at stress levels between 26,000 and 32,000 psi (stress decrements 6 to 8 per cent), can be clearly seen. It can also be observed that the quasi-steady creep rates developed after the blocking periods are quite close to those observed at the same stress levels during the sequence of stress increments; this is to be expected since the step test is still a constant-load creep test at which essentially $\dot{\sigma} = 0$. Since under conditions of steady-state creep a decrease of the constant stress by only a few per cent reduces the flow rate to zero for a substantial period, after which a gradual increase takes place towards a new steady-state condition associated with the reduced stress, the continuous decrease of the stress ($\dot{\sigma} < 0$) towards a relatively low value, characteristic of stress-relaxation processes, might be expected to cause a reduction of slip throughout the whole relaxation period, as the continuous decrease of stress seems to counteract the establishment of conditions of steady-state creep depending partially on crystal slip. The reduction will probably be most pronounced during the early stages of relaxation where $\dot{\sigma}$ differs significantly from zero and vanish as $\dot{\sigma} \rightarrow 0$. This conclusion seems to be supported by the fact that where discontinuous deformation is observed under constant stress, the "waiting time" between deformation jumps increases with time, suggesting a gradual "exhaustion" of mobile dislocations or other imperfections (13). Under decreasing stress such exhaustion would probably be more rapid; only a small fraction of the dislocations would therefore be mobilized during the (initial) period of relatively high stress levels, followed by early stoppage of slip.

Relaxation and Creep:

The use of the conventional relation $\dot{\sigma} = -E\dot{\epsilon}''$ for the conversion of one-dimensional creep data into relaxation data disregards the facts that creep in metals and in crystalline polymers is not simply viscous flow, as well as that technical creep tests are performed under constant load and therefore (because of the lateral contraction accompanying the extension) under slightly increasing stress ($\dot{\sigma} > 0$), while stress-relaxation represents an unloading process ($\dot{\sigma} < 0$). Hence the plastic part of the creep (slip and crystal-fragmentation) is retarded or blocked during this process. The neglect of creep recovery in the conventional conversion introduces a further source of discrepancy, increasing, for a selected criterion of comparisons, the difference between creep rate and relaxation rate.

However, even without considering the slip-blocking effect, the introduction of the simple equation $\epsilon'' = \epsilon''(\sigma, t)$ as an equation of state leads to the relaxation equation

$$d\epsilon'' = \left(\frac{\partial\epsilon''}{\partial\sigma}\right)_t \cdot d\sigma + \left(\frac{\partial\epsilon''}{\partial t}\right)_\sigma dt = -\frac{1}{E}\,d\sigma$$

$$-\frac{d\sigma}{dt} = +\left(\frac{\partial\epsilon''}{\partial t}\right)_\sigma \Big/ \left[\left(\frac{\partial\epsilon''}{\partial\sigma}\right) + \frac{1}{E}\right]$$

$$= E''\left(\frac{\partial\epsilon''}{\partial t}\right)_\sigma = E''\dot{\epsilon}'' \quad (4)$$

where the modulus of the true isochronous stress-strain diagram

$$E\Big/\left(1 + E\frac{\partial\epsilon''}{\partial\sigma}\right) = E'' < E.$$

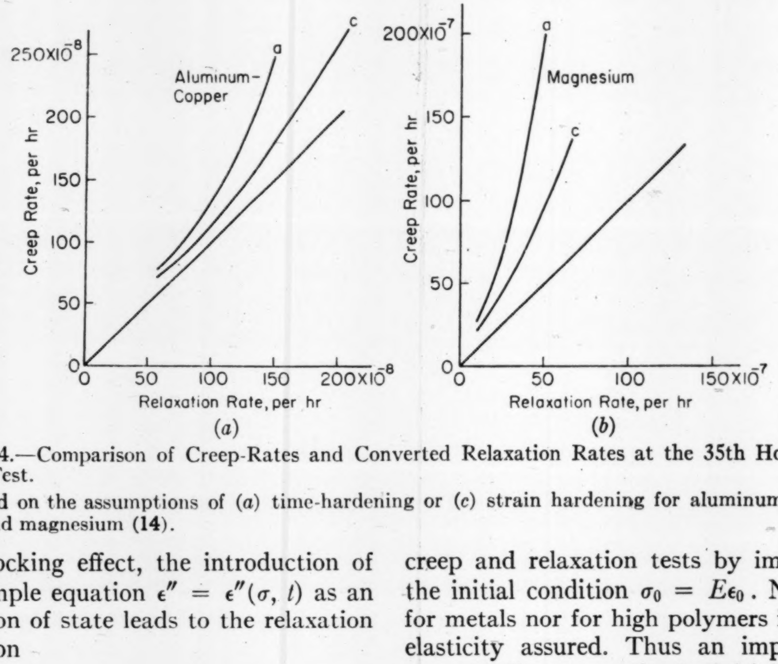


FIG. 4.—Comparison of Creep-Rates and Converted Relaxation Rates at the 35th Hour of a Creep Test.

Based on the assumptions of (*a*) time-hardening or (*c*) strain hardening for aluminum-copper alloy and magnesium (**14**).

Hence the (negative) relaxation rate is smaller than $E\dot{\epsilon}''$. Figure 4(*a*) illustrates the results of such a comparison for an aluminum-copper alloy and Fig. 4 (*b*) for magnesium at the 35th hr of the test (**14**), for two different criteria of comparison $\dot{\epsilon}'' = f_1(\sigma, t)$ and $\dot{\epsilon}'' = f_2(\sigma, \epsilon'')$.

There are, moreover, the differences arising from the application of the initial strain ϵ_0. Only perfect elasticity of the material during this application permits the direct correlation at time $t = 0$ of creep and relaxation tests by imposing the initial condition $\sigma_0 = E\epsilon_0$. Neither for metals nor for high polymers is such elasticity assured. Thus an important cause of discrepancy in the initial stages of relaxation and converted creep tests are initial strains exceeding the elastic limit.

The solution of the conventional conversion equation $\dot{\sigma} = -E\dot{\epsilon}''$ for incompressible linear viscous flow $\dot{\epsilon}'' = \sigma/\lambda$, where $\lambda = 3\eta$ denotes the coefficient of viscous traction while η is the coefficient of shear viscosity, is $\sigma = \sigma_0 \exp[-(t/\tau)]$; expressing t in terms of the relaxation time $\tau = \lambda/E$, a constant ratio of initial stress σ_0 and residual stress σ is obtained for any dimensionless time t/τ. The experimental observation (15) that for any specified time the ratio σ_0/σ is not constant but increases with the initial stress illustrates the joint effect of the nonlinearity of creep and the existence of creep-recovery (after-effect). The second effect is significant only during the early stages of relaxation tests; its separation, however, requires very careful intermittent unloadings during the creep tests. Such observations are extremely rare (9,18); isolated reports of close agreement of relaxation tests and converted creep tests (16) disregarding creep-recovery suggest either particular test conditions under which after effect is absent or too low sensitivity of the automatic recording devices.

The fact that the inelastic strain in relaxation tests is usually quite small ("micro-creep") makes the assumption of the existence of an equation of state not unreasonable, at least in thermally stable materials. Neglecting higher than first time derivatives, but considering the effect of time, the assumption of the existence of such an equation between the mechanical variables

$$\sigma = F(\dot{\sigma}, \epsilon'', \dot{\epsilon}'', t)$$

or

$$\dot{\epsilon}'' = f(\dot{\sigma}, \sigma, \epsilon'', t)$$

leads to the alternative generalized differential relations

$$d\sigma = \frac{\partial\sigma}{\partial\epsilon''}\,d\epsilon'' + \frac{\partial\sigma}{\partial\dot{\epsilon}''}\,d\dot{\epsilon}'' + \frac{\partial\sigma}{\partial\dot{\sigma}}\,d\dot{\sigma} + \frac{\partial\sigma}{\partial t}\,dt$$

$$= A\,d\epsilon'' + B\,d\dot{\epsilon} + C\,d\dot{\sigma} + D\,dt\,..(5)$$

where A may be interpreted as a variable (isochronous) modulus of strain-hardening, B as a variable coefficient of viscosity, C as a variable stress-relaxation time and D as a rate of time softening ($D < 0$) or time hardening ($D > 0$), expressed by the rate of spontaneous stress decrease or increase, and

$$d\dot{\epsilon}'' = \frac{\partial\dot{\epsilon}''}{\partial\dot{\sigma}}\,d\dot{\sigma} + \frac{\partial\dot{\epsilon}''}{\partial\sigma}\,d\sigma + \frac{\partial\dot{\epsilon}''}{\partial\epsilon''}\,d\epsilon'' + \frac{\partial\dot{\epsilon}''}{\partial t}\,dt$$

$$= a\,d\dot{\sigma} + b\,d\sigma + c\,d\epsilon'' + d\,dt\,..(6)$$

where a may be interpreted as an inverse tangent modulus, b as a variable coefficient of fluidity, $1/c$ as function of time, and d as a coefficient of time-dependent (temperature-induced) flow.

For the creep test for which $d\sigma/dt = 0$, Eq 5 is transformed into the differential equation for the creep-strain

$$A\,\frac{d\epsilon''}{dt} + B\,\frac{d^2\epsilon''}{dt^2} \pm D = 0\,\ldots\ldots(7)$$

For relaxation for which $d\epsilon = d\epsilon'' + d\epsilon' = 0$, where ϵ' denotes the elastic strain, $\epsilon' = \sigma/E$, Eq 6 is transformed into

$$(a + 1/E)\,\frac{d^2\sigma}{dt^2} + b\,\frac{d\sigma}{dt} + c\,\frac{d\epsilon''}{dt} + d = 0\,..(8)$$

With the approximations $(a + 1/E) = a'(\sigma)$, $b = b(\sigma)$, $c = c(\sigma)$, the assumption $d = 0$ and $\dot{\epsilon}'' \sim f(\sigma)$, Eq 8 represents the stress-relaxation equation

$$a'\,\frac{d^2\sigma}{dt^2} + b\,\frac{d\sigma}{dt} + cf(\sigma) = 0\,\ldots\ldots(9)$$

of a nonlinear incompressible viscoelastic material which, as the comparison with Eq 8 shows, is an approximation to the real material defined by $\dot{\epsilon}'' = f(\sigma, \dot{\sigma}, \epsilon'', t)$. The conventional rule for the conversion of creep into relaxation data which is obtained from Eq 9 by the further approximation $a' = 0$, $c/b = \text{constant} = E$ is thus revealed as a crude oversimplification.

MATHEMATICAL REPRESENTATION OF STRESS-RELAXATION

Mechanical Model of Uniaxial Relaxation:

The analysis of viscoelastic response with the aid of mechanical models provides an insight into the interaction between the individual phenomena making up the observed response, particularly creep and after-effect, that cannot be obtained from the direct consideration of the response equations.

The model that produces the creep Eq 7 under sustained stress and the relaxation Eq 9 under sustained strain is a four-parameter model obtained by the coupling in series of a linear Kelvin model with a Maxwell model with nonlinear viscosity (Fig. 5(*a*)), or of that of a linear standard solid with a nonlinear viscous dashpot (Fig. 5(*b*)) (**17**). Its equation is of the form

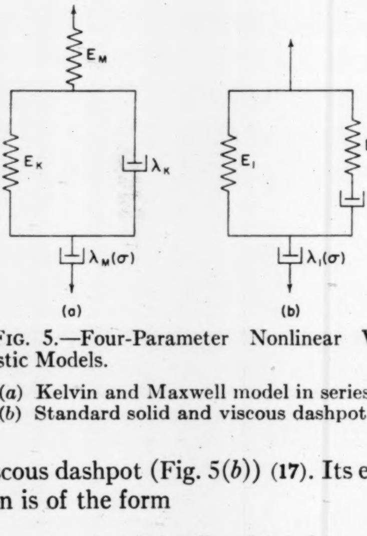


FIG. 5.—Four-Parameter Nonlinear Viscoelastic Models.

(*a*) Kelvin and Maxwell model in series;
(*b*) Standard solid and viscous dashpot.

$$a_0\sigma + a_1\dot{\sigma} + a_2\ddot{\sigma} = b_1\dot{\epsilon} + b_2\ddot{\epsilon} \quad \ldots\ldots (10)$$

with the parameters according to Fig. 5(*a*)

$$a_0 = E_k/\lambda_M(\sigma) + \lambda_k \partial/\partial t[1/\lambda_M(\sigma)]$$

$$a_1 = 1 + E_k/E_M + \lambda_k/\lambda_M(\sigma);\ a_2 = \lambda_k/E_M$$

$$b_1 = E_k\,;\ b_2 = \lambda_k \quad \ldots\ldots (11)$$

Because of the microcreep involved in stress-relaxation it is assumed that the nonlinearity is concentrated in the viscous flow part of the response while the aftereffect is linear. For a linear Kelvin model, the parameters b_1 and b_2 are true constants.

It is convenient to introduce the nonlinearity in the form of a simple power function. Thus, $\lambda_M(\sigma) = (B\sigma^{n-1})^{-1}$ so as to make the equation of the nonlinear dashpot $\dot{\epsilon}_M = \sigma/\lambda_M = B\sigma^n$. With this assumption

$$a_0 = BE_k\sigma^{(n-1)}\left[[1 + \tau_k{}^{(n-1)}\frac{d}{dt}\ln\sigma\right]$$

$$\text{with } \tau_k = \lambda_k/E_k$$

$$a_1 = 1 + E_k/E_M + B\lambda_k\sigma^{(n-1)}$$

$$a_2 = \lambda_k/E_M\,;\quad b_1 = E_k\,;\quad b_2 = \lambda_k \quad \ldots\ldots (12)$$

While Eq 10 for relaxation, $\epsilon = \epsilon_0$, with coefficients according to Eq 12 can only be solved by numerical methods or by linearization over short intervals, it is instructive to compare the solution of the linearized equation with that of the conventional transformation equation of creep to relaxation

$$\dot{\epsilon}'' = \dot{\epsilon} - \dot{\epsilon}' = f(\sigma)$$

$$\text{or}\quad \dot{\epsilon} = \dot{\sigma}/E + f(\sigma) = 0 \quad ..(13)$$

under the same assumption $n = 1$ or $f(\sigma) = \sigma/\lambda_M = B\sigma$ where $B = 1/\lambda_M$ denotes the fluidity of linear viscous creep. With

$$a_0/a_2 = (\tau_M\tau_k)^{-1} = a_0',$$

$$a_1/a_2 = \tau_m^{-1}(1 + \lambda_M/\lambda_k + \tau_M/\tau_M) = a_1',$$

$$b_1/a_2 = E_M\tau_k^{-1} = b_1$$

and

$$b_2/a_2 = E_M$$
$$= b_2'$$

the linearized Eq 10

$$\ddot{\sigma} + a_1'\dot{\sigma} + a_0'\,\sigma = b_1'\dot{\epsilon} + b_2'\ddot{\epsilon} \quad \ldots\ldots (14)$$

can be solved with the aid of Laplace transforms for a strain ϵ_0 suddenly applied at time $t = 0$ and sustained (unit-step strain). The transforms of the first and second differential of the unit-step strain $\epsilon_0 U(t)$ being, respectively, ϵ_0 and

$p \cdot \epsilon_0$, the transformed Eq 14 is of the form

$$p^2\bar{\sigma} + a_1'p\bar{\sigma} + a_0'\bar{\sigma} = b_1'\epsilon_0 + b_2'p\epsilon_0 \quad (15)$$

and therefore

$$\bar{\sigma} = (b_1' + b_2'p)\epsilon_0/(a_0' + a_1'p + p^2) \quad (16)$$

Introducing $4\lambda^2 = (a_1'^2 - 4a_0')$ and $\beta_{1,2} = -\frac{1}{2}a_1' \pm \lambda$, the transformed relaxation Eq 16 can be expanded in terms of partial fractions

$$\bar{\sigma} = C_1/(p - \beta_1) - C_2/(p - \beta_2) \quad (17)$$

where:

$$C_1 = \epsilon_0(b_1' + \beta_1 b_2')/2\lambda$$

$$\text{and} \quad C_2 = \epsilon_0(b_1' + \beta_2 b_2')/2\lambda$$

The inverse transform of Eq 17 is the relaxation equation of the four-parameter linear viscoelastic medium

$$\begin{aligned}\sigma(t) &= [C_1 \exp(\beta_1 t) - C_2 \exp(\beta_2 t)] \\ &= \exp(-\tfrac{1}{2}a_1't)[(C_1 - C_2)\cosh(\lambda t) \\ &\quad + (C_1 + C_2)\sinh(\lambda t)] \quad (18)\end{aligned}$$

which compares with the solution of the linearized conventional Eq 13

$$\sigma(t) = \epsilon_0 E \exp(-t/\tau_M) \quad (19)$$

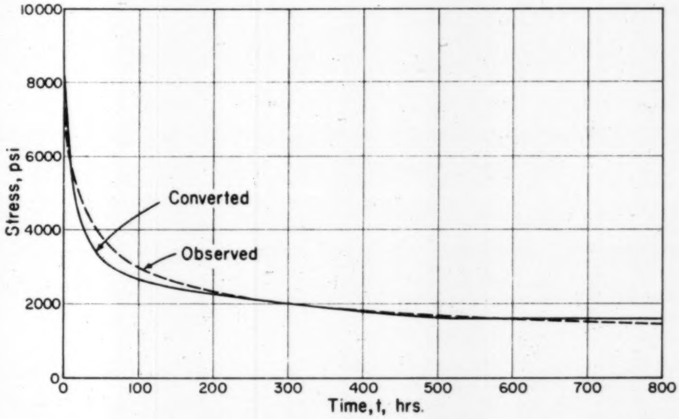


FIG. 6.—Comparison of Observed and Converted Relaxation Curves Using Nonlinear Four-parameter Model (Fig. 5(*a*)) (**19**).

It is obvious that the relaxation function $\sigma(t)$ according to Eq 18 differs significantly from the simple converted creep function Eq 19. Since the expression in brackets of Eq 18 increases with increasing time t, the relaxation process is slower than the simple exponential relaxation resulting from Eq 19.

In the approximate solution of the nonlinear Eqs 10 and 13 by linearization over short ranges, the basic differences in the solution of the linear equations over the various ranges will produce significant differences in the nonlinear relaxation curves. The application of Eq 10 to the interpretation of results of creep and relaxation tests on copper obtained by Davis (**18**) has produced agreement between the observed and predicted relaxation that could not be obtained by the simple creep-conversion Eq 13 (Fig. 6) (**19**). The application of Eq 10 is based on the simplifying assumption that the nonlinearity in the creep, $\sigma = 0$, and in the relaxation process, $\dot{\sigma} < 0$, is identical. An extensive series of tests is being performed at Columbia University to test the validity of this assumption (**19**).

Stress-Relaxation in the Continuum:

One-dimensional stress-relaxation as observed in standard relaxation tests does not represent the rather complex stress-relaxation processes within the continuum. This complexity arises from the various types of restraints on the displacements along the boundaries that can be imposed in a three-dimensional problem; such variation may involve the selection of the parts of the boundary along which restraints (sustained displacements) are imposed, as well as the selection of the form of those restraints in terms of the displacement components that are imposed and kept constant and thus of the strain-rate components that are zero.

The definition of relaxation in the continuum cannot refer to an individual stress component alone but only to an invariant combination of all stress components representing a measure of the "stress intensity," since the decrease of an individual stress component may still be accompanied by an increase of the over-all stress intensity. Assuming the absence of volume viscosity, the stress intensity that is subject to relaxation can be expressed as a function of the second invariant J_2' of the stress deviation (**17**, p. 240). $s_{ij} = \sigma_{ij} - \delta_{ij}p$, where $\delta_{ij} = 1$ for $i = j$ and $\delta_{ij} = 0$ for $i \neq j$:

$$J_2' = \tfrac{1}{2}s_{ij}s_{ij} = \tfrac{1}{2}(s_1^2 + s_2^2 + s_3^2)$$
$$= \tfrac{1}{6}[(\sigma_1 - \sigma_2)^2 + (\sigma_2 + \sigma_3)^2 + (\sigma_3 + \sigma_1)^2] \quad (20)$$

where σ_1, σ_2, σ_3 and s_1, s_2, s_3 are, respectively, the principal components of stress and stress deviation. An increase of the stress intensity is always accompanied by an increase of "effective" inelastic strain and thus by a positive rate of strain work. A decreasing stress intensity can still be associated with a positive rate of applied strainwork; it is essentially a matter of definition whether stress-relaxation is identified with "unloading" for which $dJ_2'/dt < 0$, or only with that lower limit of unloading for which also the applied rate of irrecoverable strain work is zero.

Equation 10 can be generalized by combining the six equations for the six stress-and-strain components in shear and their time derivatives

$$\alpha_0 s_{ij} + \alpha_1 \dot{s}_{ij} + \alpha_2 \ddot{s}_{ij} = \beta_1 \dot{e}_{ij} + \beta_2 \ddot{e}_{ij} \quad (21)$$

with the equation of elastic compressibility

$$p = K\epsilon_v \quad (22)$$

where $3p = (\sigma_1 + \sigma_2 + \sigma_3)$ and $\epsilon_v = \epsilon_1 + \epsilon_2 + \epsilon_3$ and $e_{ij} = \epsilon_{ij} - \delta_{ij}\epsilon_v/3$. The parameters are those of Eq 11 according to Fig. 6(*a*) in which E_M, E_k, $\lambda_M(\sigma)$ and λ_k have been replaced respectively by $2G_M$, $2G_k$, $2\eta_M(J_2')$ and $2\eta_k$.

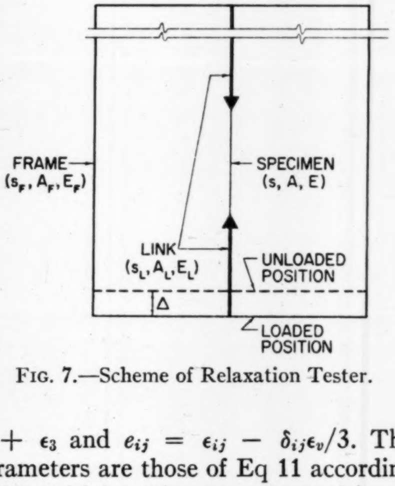


FIG. 7.—Scheme of Relaxation Tester.

In order to solve relaxation problems the six equations involving the components σ_{ij} and ϵ_{ij} obtained from Eqs 21 and 22 must be solved for the imposed strain - time functions in the various directions. To determine the relaxation of the stress intensity of the combined stresses, Eq 21 is multiplied by s_{ij} and, neglecting products of derivatives, can be written in the approximate form:

$$2\alpha_0 J_2' + \alpha_1 \dot{J}_2' + \alpha_2 \dot{J}_2'' = \beta_1 \dot{w} + \beta_2 \ddot{w} \quad (23)$$

where $\dot{w}$ denotes the applied rate of specific strain work, which is zero for true relaxation without loading in any direction. For the linearized Eq 23 a solution of the form of Eq 18 for $J_2'(t)$ can be obtained.

THE RELAXATION TEST

Theory of the Test:

Because of the elasticity of the frame and the specimen-holding links of the relaxation machine (Fig. 7) the strain ϵ of the specimen controlled through the links cannot be kept constant by directly keeping the initial deformation Δ constant but only by applying a force to do so; this force will depend on the momentary stress in the specimen, on the difference between the temperatures of the specimen T_s and the link and frame T_F, as well as on the difference in the respective coefficients of thermal expansion α_s and α_F. Denoting by s, s_L, s_F and A, A_L, A_F, respectively, the length and cross-sectional areas of specimen, links and frame and by E the elastic modulus of frame and links, the following equality exists after initial elongation Δ of specimen and links

$$(s + \Delta s \pm \alpha_s T_s s) + (s_L + \Delta s_L \pm \alpha_F T_F s_L) = \Delta + (s_F - \Delta s_F \mp \alpha_F F_F s_F)$$

Since in the undeformed state at zero reference temperature $s + s_L = s_F$, the above condition can be written in the form:

$$\epsilon = \epsilon_0 - k\sigma \pm (\alpha_s T_s - \alpha_F' T_F) \quad \ldots\ldots (24)$$

where $\epsilon_0 = (\Delta/s)$, $\alpha_F' = \alpha_F \cdot s_F + s_L)/s$, σ and ϵ the stress and strain in the specimen and

$$k = \frac{A}{E_F A_F} \frac{s_F}{s} \left(1 + \frac{A_F s_L}{A_L s_F}\right)$$

is a response characteristic of the machine and specimen combined.

In order to sustain the specimen-strain constant $\dot{\epsilon} = 0$ and therefore

$$sk\dot{\sigma} \mp s(\alpha_s \dot{T}_s - \alpha_F' \dot{T}_F) = d\Delta/dt \quad \ldots (25)$$

which implies a continuous adjustment of the imposed elongation Δ to compensate for varying stress and temperatures. Without such adjustment, the relaxation function is obtained by introducing the unit-step strain $\epsilon_0 U(t)$ into Eq 24 and solving the resulting Eq 14

$$(1 + b_2'k)\ddot{\sigma} + (a_1' + b_1'k)\dot{\sigma} + a_0'\sigma = b_1'\epsilon_0 \dot{U}(t) + b_2'\epsilon_0 \ddot{U}(t) \pm f(\Delta t) \quad \ldots (26)$$

where:

$$f(\Delta T) = b_1'(\alpha_s \dot{T}_s - \alpha_F' \dot{T}_F) + b_2'(\alpha_s \ddot{T}_s - \alpha_F' \ddot{T}_F),$$

provided that at time $t = 0$ the system is at reference temperature.

The solution of Eq 26 is clearly no longer the relaxation function of the material, but a combined response of material, testing machine and temperature of the form of Eq 18, with additional terms resulting from $f(\Delta T)$, and with parameters a_1'', and λ' depending on k.

Design Significance of Relaxation Data:

The design significance of relaxation data as obtained in current testing procedures suffers from the fact that these data reflect the combined response of the material specimen and of the machine. These conditions of elastic restraint are unrelated to the conditions in the real structures, which may differ significantly for different types of structures as well as for different parts of the same structures. Relaxation data as currently obtained therefore have rather limited design significance unless the machine effects have been completely eliminated. Since, in general, such elimination will be the exception rather than the rule, most relaxation data obtained on testing machines of current design provide not more than figures of comparison of relaxation behavior of different materials under only roughly similar conditions; the observed relaxation rates are not directly representative of relaxation rates in actual structures.

In the theoretical analysis of the relaxation test, a continuous decrease of the load of the specimen is assumed. In reality, however, most of the relaxation testing procedures necessarily involve the removal of finite load decrements; the principal difference between different designs of relaxation-test units is in the magnitude of those decrements. Tests in which the stress-adjustment unloading is made in relatively large steps presumably differ only in the "details" of this adjustment (20): the decrease of the applied load is either controlled by the limiting strain or by a constant period of loading. In the first case the load is reduced as soon as the limiting strain is attained, so that the average strain is always less than the limiting strain; in the second case the specimen is forced back to its initial strain after each loading period so that the average strain always exceeds the limiting strain. It is obvious that such details in the testing procedure will affect the resulting relaxation diagrams, since the deformation in the polycrystalline metal aggregate will reflect the differences in the various sequences of adjustment of loading to limiting strain.

Hence when the load decrements are relatively large and individual loading periods long, the relaxation test is transformed into a series of creep tests under consecutively lower load levels. Since the blocking of slip following a sudden decrease of stress is of relatively short duration, "relaxation rates" observed in such tests are likely to be higher than those observed under continuous stress-relaxation.

The observed relaxation diagrams or the relaxation strength values at specified times, defined as the residual elastic stresses at these times, will therefore reflect the testing procedures by which they have been obtained; diagrams and values obtained by procedures differing in some apparently minor detail of initial load application, load reduction or temperature control may therefore not be comparable. As, moreover, the constants $b_1'k$ and $b_2'k$ (see Eq 26) depend on the ratios of the elastic moduli of specimen and machine, which is temperature-sensitive, relaxation data, even when obtained by the same procedure on the same machine but at different temperatures, can only be compared after the machine effect has been eliminated.

Another source of discrepancy between relaxation test data and relaxation in structural elements is the effect of the loading, particularly the rate of application of the initial load in comparison to such application in actual structures. Moreover, when the initial loads or stresses are due to applied temperature differences or temperature gradients, it is unlikely that such conditions can be closely enough represented by mechanically applied initial loads under constant temperature to make the test results representative of structural behavior under stress-inducing transient temperature gradients.

CONCLUSIONS

1. The conventional procedure of conversion of creep into relaxation data with the aid of Eq 13 is unreliable. Such conversion, if possible at all, should be based on the application of Eq 10 to the interpretation of both creep and relaxation.

2. The need for extremely sensitive, very rapidly responding automatic feedback mechanisms controlled by strain gages, combined with the need for close temperature control, increases the difficulties of performing reliable relaxation

tests. Because of the strong dependence on the response of the testing machine (see Eq 26) such data are not as useful to the designer as data which would clearly reflect the stress rate effect in inelastic straining and could be obtained by experiments under linearly increasing and linearly decreasing stresses at different controlled rates. It seems simpler to construct relaxation curves of the material itself from such data than to rely on direct observation in which the variable stress rate is strongly machine controlled and therefore not clearly related to the rates of stress relaxation in the structure.

REFERENCES

(1) P. G. Nutting, "A Study of Elastic Viscous Deformation," *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 1162 (1921).

(2) F. R. Shanley, "Weight-Strength Analysis of Aircraft Structures," p. 284, McGraw-Hill Book Co., Inc., New York, N. Y., p. 284 (1952).

(3) F. J. Gillig, "Relaxation Behavior of Titanium Alloys," *WADC Technical Report*, No. 55-458, Wright Air Development Center, Part II, pp. 13–16 (1956).

(4) A. H. Cottrell, Report of the Bristol Conference on Strength of Solids, Physical Soc. (London), p. 30 (1948).

(5) B. Albrecht and A. M. Freudenthal, "Relaxation Spectra of High Polymers," *Transactions*, Am. Soc. Rheology (1958).

(6) C. Zener, "Elasticity and Anelasticity of Metals," Chicago Univ. Press, p. 126, (1948).

(7) R. W. Cahn, "Recrystallization of Single Crystals after Plastic Bending," *Journal of the Institute of Metals*, Vol. 76, p. 121 (1949).

(8) G. R. Wilms and W. A. Wood, "Mechanism of Creep in Metals," *Journal of the Institute of Metals*, Vol. 76 (8) p. 693 (1949).

(9) W. A. West, "Elastic After-Effects in Iron Wires from 20 to 550 C," *Transactions*, Am. Institute of Mining and Metallurgical Engineers, Vol. 167, p. 192 (1946).

(10) D. McLean, "Crystal Slip in Aluminum During Creep," *Journal of the Institute of Metals*, Vol. 81, p. 133 (1952/53).

(11) A. H. Cottrell and V. Ayetkin, "The Flow of Zinc Under Constant Stress," *Journal of the Institute of Metals*, Vol. 77, p. 413 (1950).

(12) R. P. Carreker, J. G. Leschen, and J. D. Lubahn, "Transient Plastic Deformation," *Transactions*, American Institute Mining and Metallurgical Engineers, Vol. 180, p. 139 (1949).

(13) H. T. Corten, M. E. Clark, and O. M. Sidebottom, "Peculiar Behavior of Steel Beams under Dead Loads that Produce Inelastic Strains," *Journal of Applied Mechanics*, Paper 51-F-4 (1951).

(14) P. Laurent and M. Eudier, "Fluage et Relaxation," *Revue de Métallurgie*, Vol. 45, p. 415 (1948).

(15) A. Nadai and J. Boyd, "Creep and Relaxation of Oxygen-Free Copper," *Proceedings*, Fifth International Congress of Applied Mechanics (1938).

(16) W. E. Trumpler, "Creep Under Positive and Negative Stress Rates," *Journal of Applied Physics*, Vol. 12, No. 3, March 1941, pp. 248–253.

(17) A. M. Freudenthal and H. Geiringer, "Mathematical Theories of the Inelastic Continuum," *Encyclopedia of Physics*, Vol. 6, p. 271, Springer, Berlin (1958).

(18) E. A. Davis, "Creep and Relaxation of Oxygen-Free Copper," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, p. A-101 (1943).

(19) D. Morecombe, M. Bieniek and A. M. Freudenthal, "Creep Under Positive and Negative Stress Rates," WADD Tech. Report (1961) (in preparation).

DISCUSSION

Mr. R. M. Goldhoff.[1]—I would first like to compliment the author on his exhaustive treatment of this difficult subject.

In the original Wright Air Development Center report of several years ago he concluded, as he has in this presentation, that current uniaxial tension relaxation tests might well be dispensed with in favor of a simpler and more indicative test to evaluate stress rate effects. I want to be clear on what he is proposing. Is he recommending constant stress rate testing at both increasing and decreasing rates in conjunction with the linear equation shown for the proper prediction of relaxation or conversion from creep to relaxation?

Mr. A. M. Freudenthal (*author*).—This is essentially what I am proposing at present, since we do not have any information on the stress-rate effect. However, I believe the non-linear equation rather than its simplified linear form might have to be used in conversion from creep under constant (including zero) stress rate to relaxation.

---

[1] Applied Research Metallurgist, General Electric Co., Schenectady, N. Y.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ACOUSTICAL FATIGUE

Supersonic flight and the development of high-thrust jet propulsion has tremendously increased the importance of the study of acoustical fatigue. Many of the problems are new, and these along with discussions of better-known ones, were the subjects of the symposium on acoustical fatigue.

B. J. Lazan presided at the symposium, sponsored by Committee E-9 on Fatigue and presented in five papers presented at the Seventh Session of the Sixty-third Annual Meeting held June 26 to July 1 at Atlantic City, N. J.

The papers presented discussed the various properties of materials important in acoustical fatigue, effect of these properties on acoustical fatigue life, and simulated environmental testing procedures for appraising acoustical fatigue properties.

The papers presented are as follows:

Introduction—B. J. Lazan
Material Properties that Affect Acoustical Fatigue Life, and the Role of Damping—W. J. Trapp and B. J. Lazan
Prediction of Acoustic Fatigue Life—H. C. Schjelderup
Discussion of Fatigue of Structural Metals—A. M. Freudenthal
The Fatigue Damaging Effect of a Random Load—Waloddi Weibull
Experimental Techniques and Equipment for Acoustical Fatigue Research and Development—D. M. Forney, Jr.

These papers, along with the discussions, were combined in *ASTM Special Technical Publication No. 284* entitled "Symposium on Acoustical Fatigue."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SHEAR AND TORSION TESTING

The objectives of this symposium were (1) to obtain detailed descriptions of new or existing shear and torsion test methods as used on a wide variety of materials, (2) to determine the test variables in these test methods and to evaluate the effects of these variables on test results, (3) to compare test results using different test techniques or conditions, and (4) to describe the advantages and disadvantages of the various test techniques.

The paper and discussions in this symposium were presented at the Eighth and Twelfth Sessions of the Sixty-third Annual Meeting of the Society, held on June 28, 1960, in Atlantic City, N. J. The symposium was sponsored by Committee E-1 on Methods of Testing.

Mr. R. W. Fenn, Dow Metal Products Co., served as Symposium Chairman and presided over the Eighth Session. Mr. R. B. Clapper, Lockheed Aircraft Corp., acted as chairman for the Twelfth Session.

The paper by L. R. Lunsford entitled "Bonded Metal-to-Metal Shear Testing" is also appearing in the Symposium on Adhesion and Adhesives ASTM *Special Technical Publication No. 271.* The papers by Eugene L. Bryan and by M. E. Raville, W. B. Bickford and D. A. Huber listed below were not presented at the symposium.

The papers included in this symposium are:

Introduction—Raymond W. Fenn
Maximum Shear Strain Measurements and Determination of Initial Yielding by Use of the Photoelastic Coating Technique—Felix Zandman
Stress Distribution in Single-Shear Sheet-Specimens—C. S. Yen
Design and Development of a Tensile-Loaded Shear Specimen—E. J. Zapel
Load Transfer in Spot Welds: A Preliminary Investigation with Photoelastic Coating—N. A. Freytag
Bonded Metal-to-Metal Shear Testing—L. R. Lunsford
On the Lap-Joint Shear Test for Metal-to-Metal Bonds—Raymond S. Shane
Preparation of Metal Surfaces for Adhesive Bonding—Raymond S. Shane
Shear and Torsion Testing of Wood, Plywood, and Sandwich Constructions at the U. S. Forest Products Laboratory—W. G. Youngquist and E. W. Kuenzi
A Dynamic Test for Determining Shear Modulus of Sandwich Core Materials—M. E. Raville, W. B. Bickford, and D. A. Huber
Photoelastic Evaluation of the Panel Shear Test for Plywood—Eugene L. Bryan
A Technique for Torsional Testing of Refractory Materials at Temperatures up to 2800 C—C. E. Waller and M. L. Stehsel
Shear and Torsion Testing of Solid Materials—A Critical Discussion—R. B. Clapper.

These papers with discussion, with the exception of the paper by N. G. McCrum, were issued as *ASTM Special Technical Publication No. 289* entitled "Symposium on Shear and Torsion Testing."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON METHODS OF METALLOGRAPHIC SPECIMEN PREPARATION

The papers presented in this symposium include both reviews of established methods and reports of new techniques, many of which were developed for preparation of some of the newer metals, alloys, and ceramic materials used in aerospace and nuclear applications.

ASTM Committee E-4 on Metallography sponsored this symposium, which was held during the Ninth and Thirteenth Sessions of the Sixty-third Annual Meeting of the Society, June 28, 1960.

Mr. G. A. Ellinger, National Bureau of Standards, served as symposium chairman and presided over the morning session, while Mr. L. L. Wyman, National Bureau of Standards, presided over the afternoon session.

The papers included in the symposium are as follows:

Introduction—Mary R. Norton
Mechanical Polishing of Metallographic Specimens—Harold S. Link
Use of Diamond Abrasives in Metallographic Problems—E. C. Olden
Present Methods of Metallographic Specimen Preparation for Retention and Identification of Inclusions in Steel—C. F. Brandenburg
Simplified Metallographic Techniques for Nuclear Reactor Materials—F. M. Cain, Jr.
Automatic Polishing of Metallographic Specimens—R. L. Anderson
Metallographic Specimen Preparation by Vibratory Polishing—P. Rothstene and F. R. Turner
Experience with Vibratory Polishers and Design for Hot-Cell Application—E. J. Long, Jr., J. T. Meador, and R. J. Gray
Techniques for Controlling Surface Pitting in Polishing Ceramic Materials—R. B. Snow
Summary—L. L. Wyman

These papers with discussion, were issued as *ASTM Special Technical Publication No. 285* entitled "Symposium on Methods of Metallographic Specimen Preparation."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON LOW-TEMPERATURE PROPERTIES OF HIGH-STRENGTH AIRCRAFT AND MISSILE MATERIALS

Knowledge of the mechanical properties of structural alloys at cryogenic temperatures has become of great importance to the national aerospace program, both because of the extremely high altitudes attained and because of the use of such propellants as liquid oxygen and liquid hydrogen.

To meet this need, the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals sponsored this Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials. The mechanical properties of high-strength structural alloys at temperatures down to −423 F are covered in the papers in this symposium, with emphasis on sheet alloys, welded joints, and resistance to brittle fracture.

The symposium was held during the Twenty-seventh, Thirtieth, and Thirty-second Sessions of the Sixty-third Annual Meeting of the Society, on Thursday, June 30, 1960.

Mr. James F. Watson, Convair-Astronautics Division of General Dynamics Corp., served as Symposium Chairman and presided over the morning session. Mr. C. L. Clark, Timken Roller Bearing Co., presided over the afternoon session, and Mr. T. N. Armstrong, International Nickel Co., Inc., over the evening session.

The papers included in the symposium are:

Introduction—James F. Watson

Sharp-Notch Behavior of Some High-Strength Sheet Aluminum Alloys and Welded Joints at 75, −320, and −423 F—M. P. Hanson, G. W. Stickley, and H. T. Richards

Some Low-Temperature Properties of Aluminum Alloy Weldments—W. R. Lucas and C. E. Cataldo

Thermal Cycling Under Constant Load to Low Temperatures of Aluminum and Magnesium Alloys—V. Weiss, G. T. Schaeffer, A. Saule, and G. Sachs

Low-Temperature Properties of Cast and Wrought Magnesium Alloys—Raymond W. Fenn, Jr.

Some Mechanical Properties of Magnesium Alloys at Low Temperatures—R. P. Reed, R. P. Mikesell, and R. L. Greeson

Sharp-Edge-Notch Tensile Characteristics of Several High-Strength Titanium Sheet Alloys at Room and Cryogenic Temperatures—G. B. Espey, M. H. Jones, and W. F. Brown, Jr.

Materials for Use at Liquid Hydrogen Temperature—John H. Belton, Larry L. Godby, and Barrett L. Taft

Effect of Stress Concentration on Tensile Strength of Titanium and Steel Alloy Sheet at Various Temperatures—George Sachs and John G. Sessler

Mechanical Properties of High-Strength 301 Stainless Steel Sheet at 70, −320, and −423 F in the Base Metal and Welded Joint Configuration—James F. Watson and J. L. Christian

Effect of Zerolling on Properties of Modified Type 347 Stainless Steel—Charles R. Mayne
Properties of Some Precipitation-Hardening Stainless Steels and Low-Alloy High-Strength Steels at Very Low Temperatures—J. E. Campbell and L. P. Rice
Low-Temperature Properties of Cold-Rolled AISI Types 301, 302, 304ELC, and 310 Stainless Steel Sheet—James F. Watson and J. L. Christian
Effect of Carbon Content on the Notch Properties of 43XX-Vanadium Modified and 5 per cent Chromium Sheet Steels—E. P. Klier
Summation—A. Hurlich

These papers with discussion were issued as *ASTM Special Technical Publication No. 287* entitled "Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials."

THE REPRODUCIBILITY OF FLEXURE AND COMPRESSION TESTS FOR DETERMINING THE STRENGTH OF CEMENT*

By M. F. Kaplan[1]

Synopsis

An investigation has been made to ascertain the "within-batch," "batch-to-batch" and "between-operator" reproducibility of flexure and modified cube compression tests on hand-mixed and compacted mortar specimens. The investigation involved more than 2000 tests. The results are compared with those of the Cement Statistical and Technical Assn. (Cembureau) tests, in which the mortar test specimens are mechanically mixed and molded, the British Standard compression tests on concrete and vibrated mortar cubes, and the ASTM methods of test for the tensile and compressive strength of hydraulic cement mortars.

ASTM Specification for Portland Cement (C 150)[2] states that when the type of strength test required by the purchaser is not specified, then the results of the briquet test for tensile strength (ASTM Method C 190)[3] shall govern. The briquet test for tensile strength has been severely criticized and has been replaced in many countries by compressive and flexural strength tests. It has been suggested (**1**)[4] that in the United States, flexure tests and compressive strength tests on portions of specimens broken in flexure should supplant both the tensile briquet and the compressive strength test on 2-in. cubes (ASTM Method C 109).[5]

One of the most important requirements of a strength test for cement is its reproducibility (**2**), and tests have been made to ascertain the within-batch, batch-to-batch, and between-operator reproducibility of flexure and modified cube compression tests on hand-mixed and compacted mortar specimens.

Experimental Details

An analysis (**3**) of factors affecting methods of determining the strength of cement led to the adoption of test procedures which have been fully described elsewhere (**4**). Wherever applicable and except where otherwise stated the requirements of British Standard 12:1958 for portland cement[6] were followed. Briefly the test procedures were:

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Head of the Civil Engineering Division, Council for Scientific and Industrial Research, Pretoria, South Africa.

[2] Standard Specification for Portland Cement (C 150 – 59), 1958 Book of ASTM Standards, Part 4, p. 1.

[3] Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars (C 190 – 59), 1959 Supplement to Book of ASTM Standards, Part 4, p. 16.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

[5] Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C109 – 58), 1958 Book of ASTM Standards, Part 4, p. 124.

[6] Portland Cement (Ordinary and Rapid Hardening), British Standards Institution, British Standard 12: 1958 (1958).

(*a*) Mix proportions were 1 cement:3 sand:0.5 water by weight. Graded sand was used (see Fig. 1).

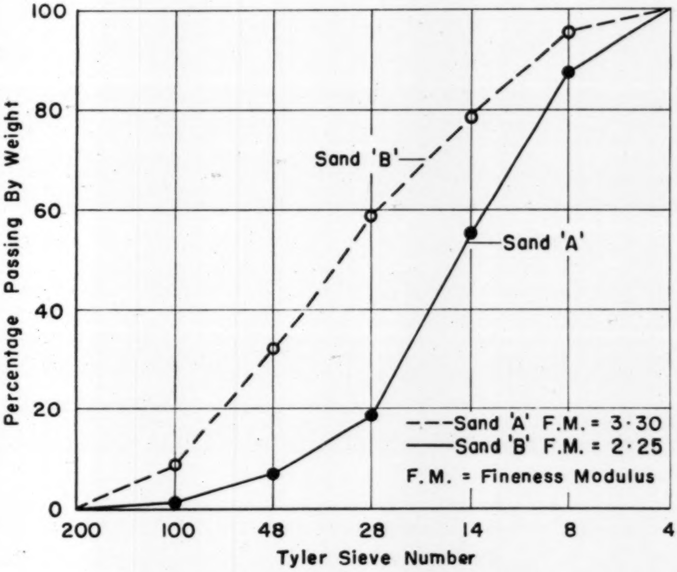


FIG. 1.—Grading Curves for the Sands Used.

TABLE I.—AVERAGE WITHIN-BATCH COEFFICIENTS OF VARIATION.

Series	Cement	Sand	Operator	Age at Test, days	Flexure Test: Number of Batches	Flexure Test: Number of Specimens per Batch	Flexure Test: Average Coefficient of Variation, per cent	Compression Test: Number of Batches	Compression Test: Number of Specimens per Batch	Compression Test: Average Coefficient of Variation, per cent
1	A	A	1	7	21	6	5.7	14	12	3.3
2	B	A	1	7	15	6	4.6	15	12	3.2
3	B	A	2	7	15	6	4.6	15	12	3.4
4	B	A	3	7	4	6	4.7	4	12	2.9
5	B	A	4	7	4	6	4.3	4	12	3.4
6	silo	B	4	3	13	3	3.2	13	6	2.8
7	silo	B	5	3	14	3	5.8	14	6	2.9
8	silo	B	4	7	14	3	4.4	14	6	3.1
9	silo	B	5	7	15	3	4.2	15	6	2.7
10	C	B	1	7	16	6	4.0	16	12	4.1
11	C	B	2	7	12	6	4.6	12	12	4.1
12	C	B	1	7	5	6	3.7	5	12	4.7

(*b*) The mixture of cement and sand was mixed dry for 1 min and then with water for 4 min.

(*c*) The nominal size of the test specimens was $1\frac{1}{2}$ by $1\frac{1}{2}$ by 8 in.

(*d*) A steel tamper having a tamping face of 1 by 1 in. and weighing 2.2 lb (1 kg) was used to consolidate the mortar in two layers. Each layer was subjected to 24 strokes of the tamper applied in three rounds of 8 strokes each. For each stroke, the tamper was raised about 1 in. above the mortar level and allowed to drop under its own weight.

(*e*) In the flexure test, the prisms were tested on a span of 6 in., with center-point loading. An "Amsler" hydraulic machine for the tension testing of mortar briquets was adapted and used for all the flexure tests except those in series 10, in which an altered "Avery" briquet testing machine loaded with lead shot was used.

(*f*) Each half of the prism broken in the flexure test was subjected to a compression test, the loaded area being 1½ by 1½ in.

TABLE II.—WEIGHTED AVERAGE WITHIN-BATCH COEFFICIENTS OF VARIATION.

Test	Number of Specimens per Batch	Sand	Coefficient of Variation, per cent		
			Age		All Tests
			3 days	7 days	
Flexure.........	3	B	4.6	4.3	4.6
	6	A	...	5.0	
	6	B	...	4.2	
Compression....	6	B	2.8	2.9	3.3
	12	A	...	3.3	
	12	B	...	4.2	

The test program is shown in Table I. All tests, except series 10, were made in the same laboratory. Each cement, A, B, and C, bought on the open market, was blended and then stored in airtight containers. In series 6, 7, 8, and 9, a sample of cement was taken each day direct from a cement silo at a cement factory. Dry river sand (sand A) and pit sand (sand B) were used in series 1 to 5 and 6 to 12, respectively. The grading curves for these sands are given in Fig. 1. Five different operators carried out the tests. Six test specimens were made simultaneously each day, except in series 6, 7, 8, and 9 when three test specimens were made. Each batch of specimens was made on different days; for example in series 1 a batch of six specimens was made on each of 21 different days. All specimens were cured in water at 24 ± 1 C. Modified cube compression tests were made on each half of the specimens broken in the flexure test, excepting the first seven batches in series 1. All tests were made 7 days after making the specimens, except in series 6 and 7 when tests were made after 3 days.

The whole program involved the making of 720 test specimens and the performance of 720 flexure and 1356 modified cube compression tests.

TABLE III.—WITHIN-BATCH COEFFICIENTS OF VARIATION (CEMBUREAU TESTS).

Test	Coefficient of Variation, per cent			
	Age at Test			Average
	3 days	7 days	28 days	
Flexure.........	4.7	4.9	4.3	4.6
Compression.....	3.9	2.9	3.4	3.4
APCM concrete compression....	4.1	2.6	3.2	3.6

WITHIN-BATCH REPRODUCIBILITY

The within-batch coefficients of variation were calculated and the average of the coefficients in each series of tests determined. The average results are given in Table I.

The number of batches in each series of tests was not always the same. The weighted average coefficients were therefore calculated for those series of tests in which the number of specimens per batch, the sand used, and the age at test were the same. The results are shown in Table II.

Six-specimen batches made with sand B and tested after 7 days had an average within-batch coefficient of variation of 2.9 per cent for the compression tests and 4.2 per cent for the flexure tests. The average results for all tests indicate that the flexure tests have a greater within-batch variation (4.6 per cent) than the modified cube compression tests (3.3 per cent).

Comparison With Other Methods of Test:

It is of interest to compare the above results with those obtained in other methods of test for cement strength.

Crandall and Blaine (4) have recently reported the results of briquet tests for tensile strength in accordance with ASTM Method C 190 and in which 103 laboratories tested 12 cements. Six test specimens were made with each sample of cement. Three of the specimens were tested after 3 days and after 7 days. For samples 3 and 4, which were type I and type IA cements, the average within-batch coefficients of variation for the three-specimen batches were 4.5 and 4.6 per cent. Coefficients of variation greater than 10 per cent were frequently reported.

The British Standard vibrated mortar cube compression test[6] produces results with a within-batch coefficient of variation of between 3 and 4 per cent. For example, in 1950 the Building Research Station in England conducted an investigation on vibrated mortar cubes in which 12 laboratories took part. Tests were made at 3, 7, and 28 days on three types of cement, and the average within-batch coefficient of variation for six-specimen batches was 3.5 per cent.

The comprehensive tests reported by Walker and Bloem (5) indicate that for three-specimen batches tested at 3, 7, and 28 days, the ASTM compression test (ASTM Method C 109) gave results with average within-batch coefficients of variation of approximately 2.5 per cent. These tests were made in one laboratory by a highly skilled, specially trained, technician.

In 1949, the Cement Statistical & Technical Association (CEMBUREAU) in cooperation with the International Union of Research and Testing Laboratories (RILEM) and the International Organization for Standardization (ISO) formed a working party on cement standards to study the testing of cement. An investigation was carried out which culminated in recommendations for an international test for cement strength. The Cembureau procedure (6) consists of flexure and modified cube compression tests on mortar beams which are mechanically mixed and compacted. The average within-batch coefficients of variation obtained in the sixth series (7) of tests in the Cembureau investigation are shown in Table III. The tests were made in 12 different laboratories and there were 3 specimens per batch in the flexure tests and 6 specimens per batch in the compression tests. The average within-batch variation was 4.6 per cent in the flexure tests and 3.4 per cent in the compression tests. Using the Cembureau methods, Dutron (8) has reported average within-batch variations for 12 specimen batches tested at 56 days of 4.5 and 3.9 per cent, respectively for the flexure and compression tests.

The Cembureau investigation included a compression test on 4-in. concrete cubes suggested by the Associated Portland Cement Manufacturers (APCM) in Great Britain. This test has been accepted as an alternate test in the latest revision of the British Standard for portland cement.[6] The average within-batch variation for three-specimen batches was 3.6 per cent.

Although the number of specimens per batch and the age at test were not always the same, the above results indicate that on the average: (*a*) Within-batch coefficients of variation for flexural strength tests are of the same order as for briquet tests for tensile strength. (*b*) Within-batch coefficients of variation for compression tests are generally less than for flexural strength tests. Coefficients of

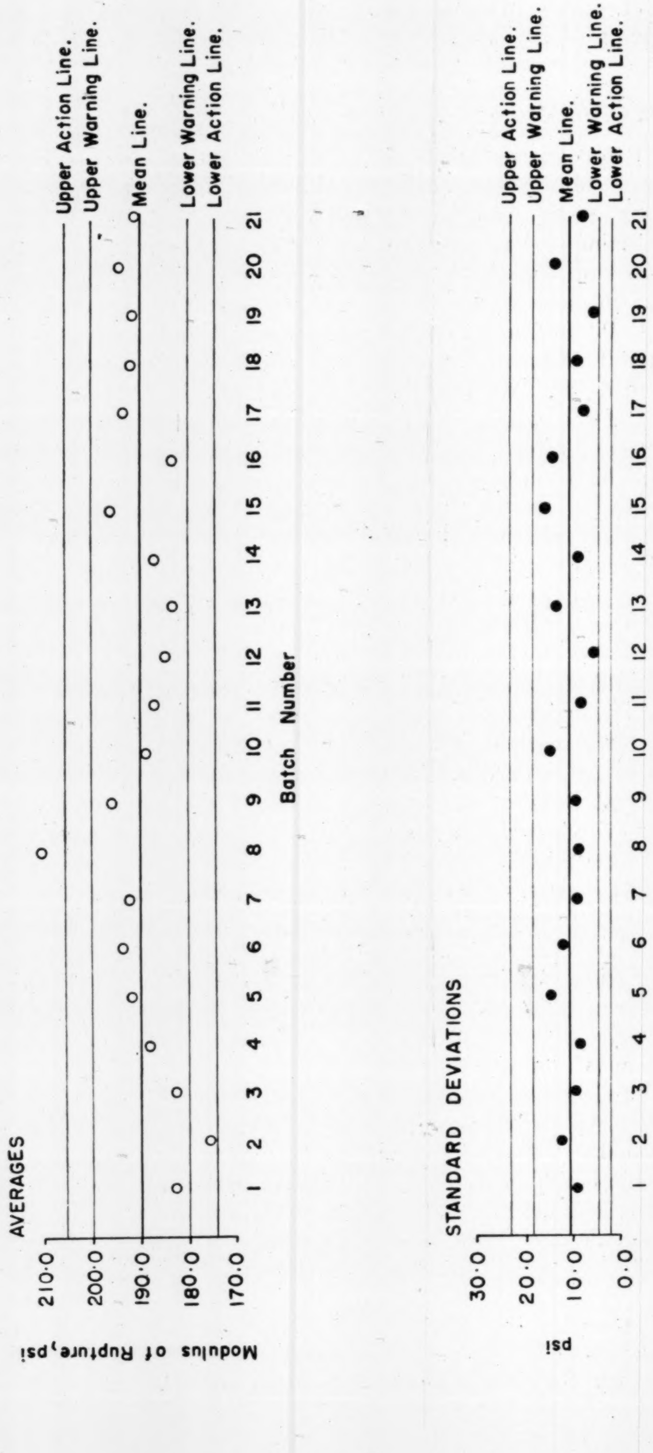


FIG. 2.—Control Charts for Flexure Tests in Series 1.

variation between 2.5 and 4 per cent may be expected in compression tests and between 4 and 5 per cent in flexure tests.

Statistical Uniformity of Within-Batch Variations:

In the above discussion the average "within-batch" coefficients of variation have been compared. The individual coefficients from which the average is determined do, however, vary. The statistical uniformity of the within-batch variations obtained in this investigation was therefore ascertained to determine whether the within-batch variability was itself under control. This was done by calculating the 1 in 40 and 1 in 1000 control limits[7] for the within-batch standard deviations and drawing the control charts. The control charts for the standard deviations in each of the 21 batches of flexure and 14 batches of compression tests in series 1 are shown in Figs. 2 and 3, respectively. The upper and lower 1 in 1000 control lines are called the upper and lower "action" lines, whilst the 1 in 40 control lines are called the upper and lower "warning" lines. According to statistical theory, if a test or process is controlled by chance and the variations are therefore due to nothing else but the influence of chance with no assignable causes disturbing the chance variation, it may be expected that approximately 1 in 40 samples will fall outside one of the warning lines and that approximately 1 in 1000 samples will fall outside one of the action lines. The test or process is then said to be statistically uniform or homogeneous and the variation is said to be statistically controlled. The number of standard deviations falling outside the warning and

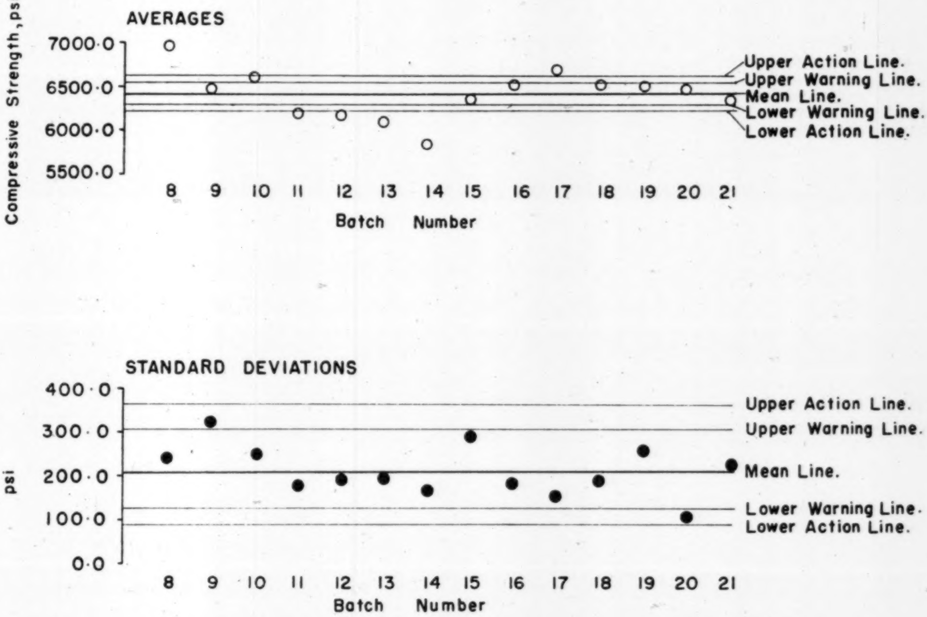


Fig. 3.—Control Charts for Compression Tests in Series 1.

---

[7] P. B. Dudding and W. J. Jennett, Quality Control Charts, British Standards Institution, British Standard 600R (1942).

action lines in series 1, 2, 3, and 11 is given in Table IV.

In three out of the four series mentioned, the within-batch standard deviations for the flexure test were under control, that is, the fluctuations in the results of tests done on the six specimens in the same batch were due entirely to chance causes. In series 11, however, one in six standard deviations fell outside the upper and lower warning lines, thus indicating that there were disturbing factors at work causing within-batch fluctuations larger than those which may be ascribed to chance. The within-batch standard deviations for the compression test were out of control in all of the four series of tests.

TABLE IV.—NUMBER OF WITHIN-BATCH STANDARD DEVIATIONS FALLING OUTSIDE THE WARNING (1 IN 40) AND ACTION (1 IN 1000) CONTROL LINES.

Series	Test	Number of Batches in Series	Number of standard Deviations	
			Between Warnings and Action Lines	Outside Action Lines
1	Flexure	21	nil	nil
2		15	nil	nil
3		15	nil	nil
11		12	2	nil
1	Compression	14	2	nil
2		15	2	nil
3		15	1	nil
11		12	2	2

In the course of an investigation (9) into the use of a South African sand for cement testing purposes, Sichel (10) carried out a similar statistical analysis on the results of the British vibrated mortar cube compression test, using the standard Leighton Buzzard sand. He also came to the conclusion that there were disturbing factors causing significant variations in the results of tests on different specimens in the same batch. When however, the proposed South African standard sand was used instead of the standard Leighton Buzzard sand there was no significant variation in the results within-batches.

Thus, although the indications are that the within-batch coefficients of variation are greater for the flexure than the compression test the variations in the results of tests on different specimens in the same batch appear to be less significant for the flexure than the compression test.

TABLE V.—BATCH-TO-BATCH COEFFICIENTS OF VARIATION.

Series	Number of Batches	Coefficient of Variation, per cent	
		Flexure Test	Compression Test
1	21[a]	3.6	3.8
2	15	2.6	3.1
3	14	4.3	6.1
11	12	2.2	3.2
Weighted average		3.2	4.1

[a] There were 14 batches in the compression test.

BATCH-TO-BATCH REPRODUCIBILITY

The reproducibility of the results of tests made on different batches of the same sample of cement was first investigated by calculating the coefficient of variation using the average strength of each batch of test specimens. The results for series 1, 2, 3, and 11 are shown in Table V. The batch-to-batch variations were always less in the flexure than in the compression test, the weighted average coefficients of variation being 3.2 and 4.1 per cent, respectively. It should also be noted that in the flexure test the batch-to-batch variations were usually less than the within-batch variations, whilst in the compression test the reverse was generally true.

In the course of the South African standard sand investigation (9) British standard vibrated mortar cube compression tests were made on six batches of the same cement. The batch-to-batch coefficient of variation was 6.4 per cent, which is also greater than the within-batch variations usually obtained with this test. It is also greater than the batch-to-batch variations for the flexure and modified cube compression tests obtained in this investigation.

TABLE VI.—NUMBER OF AVERAGES FALLING OUTSIDE THE WARNING AND ACTION CONTROL LINES.

Series	Test	Number of Batches in Series	Number of Averages	
			Between Warning and Action Lines	Outside Action Lines
1......	Flexure	21	1	1
2......		15	2	nil
3......		15	7	nil
11......		12	nil	nil
1......	Compression	14	1	6
2......		15	3	5
3......		15	2	9
11......		12	3	1

Statistical Uniformity of Batch-to-Batch Variations:

The statistical uniformity of the average strengths obtained from tests on different batches of the same cement was determined by calculating the 1 in 40 and 1 in 1000 control limits for the averages. The control charts for the average flexural and compressive strengths in series I are shown in Figs. 2 and 3, respectively. The number of averages falling outside the warning (1 in 40) and action (1 in 1000) lines in series 1, 2, 3, and 11 are given in Table VI.

With the exception of the flexural strength tests in series 11, the average flexural and compressive strengths of different batches of the same sample of cement are out of control, that is, the differences between the averages are not due to chance causes. It appears however that except in series 3, the likelihood of the batch-to-batch variations being out of control are less in the flexure than in the compression test. In seeking for an assignable cause as to why the series 3 flexure tests were out of control, it was found that because the operator in this series of tests did not always make the test specimens at the same hour each day there were differences of six hours in the age of some of the batches when tested. Although this would not affect the within-batch coefficients of variation it could have been the cause of six of the averages falling beyond the warning lines.

In the South African standard sand investigation Sichel (10) reported that when using both the standard Leighton Buzzard sand and the proposed South African standard sand, the batch-to-batch variations in the results of the British Standard vibrated mortar cube compression test could not be explained by chance fluctuations alone.

Thus in addition to the batch-to-batch coefficients of variation for the flexure test being less than for compression tests, it also appears that the differences in batch-to-batch average flexural strengths are more likely to be due to chance causes than are the compressive strengths. More work is required to establish this.

BETWEEN-OPERATOR REPRODUCIBILITY

In series 2, 3, 4, and 5, four operators working in the same laboratory made flexure and modified cube compression test specimens from the same sample of cement. Although operators 3 and 4 were experienced in the making of test specimens for the British standard tests, the making of specimens for the flexure and modified cube compression test were new to all four operators. Operator 1 had however already carried out the first series of tests. There were six test specimens in each batch. All specimens were tested by operator 1 on the same testing machine. The results of the tests are given in Table VII.

TABLE VII.—BETWEEN-OPERATOR COEFFICIENTS OF VARIATION.

Series	Operator	Flexure Test		Compression Test	
		Average Load at Failure, lb	Coefficient of Variation, per cent	Average Load at Failure, lb	Coefficient of Variation, per cent
2........	1	196	...	6650	...
3........	2	202	2.3	6815	2.4
4........	3	199	...	6400	...
5........	4	202	...	6750	...

The coefficient of variation for the average results obtained by each of the four operators was 2.3 per cent for the flexure test and 2.4 per cent for the compression test. Reference to Tables II and VI indicate that these between-operator variations are less than both the within-batch and between-batch variations when all the specimens were made by one operator. This suggests that different operators should be able to reproduce the results of flexure and compression tests on the same sample of cement.

Student's t Test:

As Wright (11) has mentioned, a statistical test which involves a quantity known as Student's *t*, may be used to determine whether an observed difference between the averages of two series of results could have been caused by the random variation of the individual test results or whether it is due to some inherent difference between the two series. This test for significance is based upon the null hypothesis which assumes that the samples in question do not exhibit any real differences. From the calculated values of *t*, the probability of the observed difference, if the null hypothesis is true, may be ascertained. If the probability is very small, then the differences may be said to be significant. The *t* values for pairs of operators were calculated and their probability values ascertained (12) (Table VIII).

TABLE VIII.—STUDENT'S *t* TEST FOR AVERAGE RESULTS OBTAINED BY DIFFERENT PAIRS OF OPERATORS.

Operators	Degrees of Freedom, ϕ	Flexure		Compression	
		t Test	Probability, per cent	*t* Test	Probability, per cent
1 and 2..................	28	1.967	5 to 10	1.268	10 to 25
1 and 3..................	17	1.097	25 to 50	1.752	5 to 10
1 and 4..................	17	2.088	5 to 10	0.577	>50
2 and 3..................	17	0.473	>50	1.664	10 to 25
2 and 4..................	17	0	100	0.237	>50
3 and 4..................	6	0.666	>50	1.007	25 to 50

Two probability values have been widely used as a limit beyond which differences are considered to be significant. If the probability is less than 5 per cent, it is generally considered that there is strong evidence of real difference, and if it is less than 1 per cent it is reasonably certain that a real difference exists. It will be seen from Table VIII that in no instance was the probability value less than 5 per cent. Although all these comparisons between operators are not independent of one another, the *t* tests show that the differences in the results of the tests made by the four operators were not significant for both the flexure and compression tests.

TABLE IX.—RELIABILITY COEFFICIENTS FOR THE MODIFIED CUBE COMPRESSION TEST.

Series	Number of "Twin" Specimens	Reliability Coefficient
1	84	0.59
2	90	0.41
3	90	0.80
6	39	0.87
7	42	0.75
10	96	0.80
11	72	0.39
Weighted average		0.64

RELIABILITY OF THE MODIFIED CUBE COMPRESSION TEST

In the modified cube compression test, two tests were performed on the same specimen—one on each half. The strengths of each half of the same specimen were compared by calculating the "reliability" coefficients. These coefficients are calculated in the same way as correlation coefficients, except that instead of comparing two different characteristics, the same characteristic of the same specimen is compared. The frequency method of calculation was adopted and the results are given in Table IX.

The weighted average coefficient of reliability was 0.64, the maximum value for any one series being 0.87. These values are less than the value of 0.90 which is generally regarded as a minimum and indicates that agreement between the results of compression tests on two halves of the same test specimen is not satisfactory. The results obtained in series 1, 2, and 3 are illustrated qualitatively in Table X; *E* denotes that the strengths of the two halves were equal, a plus (+) sign that the left half was stronger than the right half and a minus (−) sign that it was weaker than the right half. It will be seen that the results often follow a pattern; in some batches

TABLE X.—COMPARISON OF COMPRESSIVE STRENGTH OF LEFT HALF AND RIGHT HALF OF THE SAME TEST SPECIMEN.

Series 1	Series 2	Series 3
E − *E* − − −	*E* + + + + +	− − − − − −
− − − − − −	− + − − − +	+ − − − − *E*
+ − − − − −	− *E* + − *E* −	− + + − + *E*
+ + *E* + + +	+ + − + − −	− − − − + −
+ − + + *E* +	+ + − + − +	− − *E* + + −
+ − − − + −	− − − + + +	*E* − *E* + − *E*
− − − − − −	− − − *E* − +	+ + + − + +
− − − − − −	+ − − + − +	+ + + + − −
+ + + − − −	*E* − + + *E* −	+ + + + + +
+ + + + + +	− *E* + *E* − −	+ − + + + +
− + + − − +	− + + − *E* −	− − − + − +
− − + − − −	+ − − − − +	− *E* − − + −
E *E* + − − +	+ + + + − −	+ *E* + + + +
+ − − − − −	− + − − + +	+ − − − − +
	+ − + + − −	− − − − − −

E denotes left half = right half.
+ denotes left half > right half.
− denotes left half < right half.

the strength of the left half was always greater than the right half, in others it was always less. These "runs" occur too frequently to suggest that they are due to chance causes.

BETWEEN-LABORATORY REPRODUCIBILITY

Although the reproducibility of tests made in different laboratories was not investigated, it is of interest to mention the results of other investigations.

Dutron (8) has reported the results of Cembureau mortar tests made after 56 days in seven laboratories in different countries using the same Belgian sand. For tests on normal or type I cement, the between-laboratory coefficient of variation was 7.1 per cent for the flexure test and 9.0 per cent for the compression test. With rapid-hardening or type III cement, the coefficients were 7.1 and 13.5 per cent respectively for the flexure and compression tests. Test specimens made in the seven different countries were also sent to one central laboratory for testing. The coefficient of variation between the average strengths of the specimens made in the different laboratories using normal portland cement was 5.3 per cent for the flexure test and 7.6 per cent for the compression test, for rapid hardening cement the variations were 6.8 and 13.3 per cent, respectively.

In the sixth series (7) of tests in the Cembureau investigation, the average coefficient of variation between tests made in 11 laboratories was 7.3 per cent for the flexure test and 6.4 per cent for the compression test. The APCM concrete compression test, which was included in the Cembureau investigation, gave a between-laboratory variation of 9.0 per cent.

In the investigation reported by Crandall and Blaine (4), the average interlaboratory coefficients of variation, for tests on type I and type IA cements, were 8.5 per cent for the briquet test for tensile strength and 8.7 per cent for the mortar cube compression test (ASTM Method C 109).

The indications are, therefore, that the between-laboratory reproducibility of flexure tests, as measured by coefficients of variation, is at least as good, if not better than for the briquet tests for tensile strength and compression tests.

In regard to the statistical uniformity of tests made in different laboratories, it has been found (13) that for a hand-compacted mortar cube compression test the results were out of control; the differences in the average strengths obtained in the various laboratories were not due to chance causes. An analysis of the results of inter-laboratory British Standard vibrated mortar cube compression tests conducted by the Building Research Station in England in 1950 also indicated that the results were not statistically uniform. As far as it is known the results of interlaboratory briquet tests for tensile strength and flexural tests have not been similarly analyzed.

CONCLUSIONS

The main conclusions are:

1. Within-batch coefficients of variation for compression tests are generally less than for flexure tests. Coefficients of variation between 2.5 and 4 per cent may be expected in compression tests and between 4 and 5 per cent in flexure tests.

2. Although within-batch coefficients of variation are generally less for compression than for flexure tests, there is a greater likelihood in the compression test than in the flexure test that the strengths of different specimens in the same batch differ significantly from each other.

3. Batch-to-batch coefficients of variation in the flexure test were less than in the compression test.

4. In the flexure test, batch-to-batch

coefficients of variation were usually less than within-batches. For the compression test the reverse was generally true.

5. Batch-to-batch flexural strengths are less likely to differ significantly from each other than batch-to-batch compressive strengths.

6. The differences in both the average flexural strengths and the average modified cube compressive strengths obtained in tests by four different operators were not significant.

7. The agreement between the compressive strengths of two halves of the same mortar prism was not satisfactory.

REFERENCES

(1) M. A. Swayze, "A Comparison of Cement Strengths in Mortars and Concretes," ASTM BULLETIN, No. 232, Sept. 1958, p. 54 (TP 202).

(2) M. F. Kaplan, "A Critical Examination of Methods of Testing the Strength of Cement," *National Building Research Institute Bulletin*, Pretoria (South Africa), No. 15, Jan. 1957, p. 1.

(3) M. F. Kaplan, "An Analysis of Factors Affecting the Method of Determining the Strength of Cement With Particular Reference to a Proposed Transverse and a Modified Cube Compression Test," *National Building Research Institute Bulletin*, Pretoria (South Africa), No. 16, Mar. 1958, p. 47.

(4) J. R. Crandall and R. L. Blaine, "Statistical Evaluation of Interlaboratory Cement Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 1129 (1959).

(5) S. Walker and D. L. Bloem, "Variations in Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 1009 (1958).

(6) CEMBUREAU, "On the Testing of Cement.—Report of Cembureau's Working Party on Cement Standards including Method of Testing the Strength of Cement," The Cement Statistical and Technical Assn., Malmo (Sweden) (1956).

(7) CEMBUREAU, "Sixth Test Series," supplement to "On the Testing of Cement" (reference 6), The Cement Statistical and Technical Assn., Malmo (Sweden) (1958).

(8) R. Dutron, "Strengths of Cements in Plastic Mortar," *RILEM Bulletin*, The International Union of Research and Testing Laboratories (Paris), No. 35, p. 7 (1957).

(9) "South African Standard Sand for Cement Testing Purposes," *National Building Research Institute Report* (South Africa) (1949).

(10) H. S. Sichel, "A Statistical Analysis of Cement Testing Data," *National Building Research Institute Report*, on the Standard Sand Investigation, Appendix A. (South Africa) (1950).

(11) P. J. F. Wright, "Statistical Methods in Concrete Research," *Magazine of Concrete Research* (London), Vol. 5, No. 15, p. 139 (1954).

(12) O. L. Davies, Statistical Methods in Research and Production, Oliver & Boyd, London, p. 270, 2nd Edition (1949).

(13) "Interlaboratory Statistically Planned and Analyzed Investigation in connection with the Compressive Strength Test, as proposed by the South African Cement Manufacturers Assn.," South African Bureau of Standards, March 1952.

DISCUSSION

MR. MYRON A. SWAYZE.[1]—The Working Subcommittee on Strength in ASTM Committee C-1 on Cement has been interested for a long time in better methods of determining potential strengths of cement. The history of such testing is almost as old as the portland-cement industry.

It is interesting to note that the first systematic strength tests on cement of which we have knowledge were made on prisms in compression in a hydraulic press. These tests were described by A. C. Davis.[2] According to Redgrave and Spackman[3] we have the French Engineers of Bridges and Embankments to thank for briquet testing. Their studies were made in the period 1848 to 1850. John Grant of London adopted their test in 1859 and later experimented with nine other forms of briquets. In 1880 he recommended a specimen of 1 in. square section, with waist contours identical with our present specimen. J. B. Johnson[4] pointed out in 1898 the great inequalities in stresses which are set up in the minimum section of this briquet. Later, E. G. Coker[5] found even greater inequalities, with the highest stress being 1.75 times the mean stress. With our modern high-strength cements these differences can be so great that rupture at the edges of the waist can occur while the center of the section is still under compression. Hence, the briquet has never been a measure of the real tensile strength of cement.

In the period 1915 to 1917 several technical societies in the United States met under the auspices of ASTM to consider standardizing test methods and specifications for cements. At this joint conference, Subcommittee VII on Strength of ASTM Committee C-1 on Cement considered all of the data available and recommended abandonment of all briquet tests as soon as a compression test could be standardized and brought into general use. Their data on performance of neat briquets were so clear that ASTM Committee C-1 deleted specification limits for neat cement briquets in 1917. Unfortunately the mortar briquet is still used here, although it has been abandoned in many European countries and elsewhere.

As a long-time member and present chairman of Committee C-1 Working Subcommittee on Strength, I have been interested for many years in methods for determining the potential strengths of cement. It was my privilege to visit several European countries in 1938, and in 1945 I was delegated by our Government to make a survey of the German cement industry. During both of these visits I looked into cement testing methods.

Prior to World War II the common strength test was by briquets, using an earth-dry mortar mixed with 8 to 8.5

---

[1] Director of Research, Lone Star Cement Corp., New York, N. Y.

[2] A. C. Davis, "One Hundred Years of Portland Cement," Concrete Publications, Ltd., London (1924).

[3] G. R. Redgrave and Charles Spackman, "Calcareous Cements," Charles Griffin and Co., London (1924).

[4] J. B. Johnson, "The Materials of Construction," John Wiley & Sons, Inc., New York, N. Y. (1898).

[5] E. G. Coker and L. N. Filon, "A Treatise on Photoelasticity," Cambridge University Press (1931).

per cent water, in contrast with the 10 to 11 per cent which we use here in briquet mortars. In 1945 I was amazed to find that the Germans had changed completely to the Swiss flexure and compression tests on 40-mm prisms, using 50 per cent mixing water. This transition took place in 1942, when the German economy was devoted primarily to the war effort and little else. The universal reason given for the change was that the flexure and compression tests on prisms gave a more reliable index to concrete strengths than their old briquet test.

The single feature of the present European tests on prisms to which we cannot subscribe is the use of finely ground silica sand as a one third part of the sand in their 1:3 mortar mix. ASTM Committee C-1 tried out a very similar sand grading in mortars used for casting 2-in. cubes over twenty years ago and obtained phenomenally high strengths which were completely out of range of those obtainable with the same cements in normal concrete of the same water-cement ratio. In consequence, we abandoned further tests of this sort of mortar, which we referred to as "baby concrete," and I doubt if we will ever return to it.

The author does not state whether he is using this same proportion of very fine sand in the South African mortar mix. If so, then I would expect the mortar strengths are also predicting higher strengths than are found for the same cements in concrete. A more normal sand grading would give more comparable results.

Regardless of such details, however, the basic principle of running flexure tests and compression tests on the same specimens needs no argument. The two tests are closely correlated; they are realistic for the better grades of concrete; they do reflect in early strength the strengths we can expect in concrete with similar water-cement ratios. At later ages, such as 28 days, the mortar tests still exceed the strengths we find in concrete, but we can allow for these differences. It is still an approach to an ideal in the manner of testing cements for strength. It is, therefore, extremely interesting to learn that South Africa, together with most of Europe, is going to this principle for strength testing—to determine both flexural and compressive strength on the same specimen made with the same realistic water ratio. I hope to see the day when American cement testing will be done on a similar basis.

MR. M. F. KAPLAN (*author*).—The author wishes to thank Mr. Swayze for his discussion of the paper. The gradings of the sands used in the investigation are shown in Fig. 1; it will be seen that in sand A, approximately 2 per cent by weight passed the No. 100 sieve while in the case of sand B the proportion was 8 per cent.

AN X-RAY DIFFRACTION INVESTIGATION OF HYDRATED PORTLAND CEMENT PASTES*

By D. L. Kantro,[1] L. E. Copeland[1] and Elaine R. Anderson[1]

Synopsis

A number of well-hydrated pastes of a group of eight portland cements of differing compositions have been examined by means of X-ray diffraction. Data were obtained for saturated samples, dried samples, and samples dried and resaturated. The data obtained included both line positions and shifts in line position with change in specimen water content.

The diffraction results provide a qualitative identification of some of the hydration products in portland-cement pastes. Thus, it has been found that the calcium silicate hydrate phase is very much like the tobermorite obtained by the paste hydration of tricalcium silicate or beta-dicalcium silicate. The other hydration products found in cement pastes are both calcium sulfoaluminates, a hydrogarnet type hydrated calcium aluminoferrite, and a tetracalcium aluminate hydrate. This latter phase may be a solid solution with the analogous ferrite.

The low sulfate sulfoaluminate forms in cements with relatively high tricalcium aluminate contents at the expense of the high sulfate form. On the other hand, the high sulfate form remains unchanged even after long curing periods in cements low in tricalcium aluminate.

Portland cement is an intimate mixture of several components. There are in this material four major phases, namely, tricalcium silicate, or alite, beta-dicalcium silicate, or belite, tricalcium aluminate, and a calcium aluminoferrite. In cement, each of these phases is relatively well crystallized and the sizes of the phase regions are such that most of them can be distinguished by means of the light microscope. This system has been subjected to analysis by several techniques, the most recent being that of X-ray diffraction (**1**,**2**).[2] These methods rely on the fact that the compositions of the phases are much the same from cement to cement. Although the composition of the aluminoferrite phase is not the same in all cements, it may be determined in the analysis.

When portland cement reacts with water, an entirely different situation arises. Some of the products formed are in the colloidal size range, some are not well crystallized, and far more opportunity arises for formation of solid solutions that may have wide ranges of composition. Furthermore, several different stoichiometric combinations of the same substances are possible, for example, the calcium aluminate hydrates.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Research Chemist, Senior Research Chemist and Assistant Research Chemist, respectively, Basic Research Section, Portland Cement Assn. Research and Development Laboratories, Skokie, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

In the past, numerous investigations have been made in an attempt to identify the products of hydration of portland cement. However, because of the complexity of the product, conflicting results have been published at different times. By 1952, the resolution of the problem had begun, as can be seen in Steinour's review (3).

In the earlier investigations by means of X-ray diffraction, the Debye-Scherrer powder photograph method has been used for the most part. The relatively recent development of the diffractometer, however, represents a major improvement in this field. It is now possible to obtain greater resolution, so that important details heretofore unknown are revealed. The present investigation makes use of the newer diffraction technique and includes observations on the effect of state of dryness of the paste on its diffraction pattern. Certain products have been identified by X-ray means, no previous identification by any means having been made.

TABLE I.—MAJOR OXIDE CONTENTS OF THE VARIOUS CEMENTS USED.

Cement	SiO_2[a] per cent	CaO, per cent	Al_2O_3, per cent	Fe_2O_3, per cent	MgO, per cent	SO_3, per cent
15754	20.7	63.0	6.1	2.2	2.0	2.4
15622	22.4	63.8	3.9	4.2	1.4	1.6
15497	19.8	63.4	5.2	2.6	1.3	1.8
15669	27.5	63.7	2.0	1.9	1.6	1.8
LTS-17	21.4	64.8	5.4	3.1	0.9	1.7
LTS-25	22.6	61.9	4.6	4.9	2.2	1.9
LTS-31	20.3	63.3	5.0	2.1	3.2	2.2
LTS-41	22.9	59.2	4.6	5.0	2.7	2.0

[a] All oxide values were corrected as described on p. 759 of Reference (**5**).

EXPERIMENTAL PROCEDURE

Preparation of Specimens:

All specimens were pastes prepared either from members of a group of "Special Basic Research" (SBR) cements, the chemical analyses of which were reported by Powers and Brownyard (**4**), or from a group of "Long-Time Study" (LTS) cements, the chemical analyses for which were reported by Lerch and Ford (**5**). The major oxide compositions of the cements used in this investigation are given in Table I. The compound compositions of these cements have also been determined by X-ray quantitative analysis (**1**,**2**).

TABLE II.—CHARACTERISTICS OF THE VARIOUS PASTES INVESTIGATED.

Specimen	Cement	Original Water-Cement Ratio, g/g	Age
C-88	15754	0.40	11 yr
C-54	15754	0.55	11 yr
C-87	15754	0.40	7 yr
C-75	15754	0.65	7 yr
C-49	15754	0.65	5 yr
C-21	15754	0.60	3.25 yr
C66-K-11	15754	0.70	11 months
C66-O-6	15754	0.57	6 months
C61-C-17	LTS-17	0.70	17 months
C61-C-12	LTS-17	0.70	12 months
C-33-1	15622	0.60	10 yr
C-33	15622	0.60	7 yr
C-65-F-22	LTS-25	0.70	22 months
C-65-F-13	LTS-25	0.70	13 months
C-31-1	15497	0.60	10 yr
C-31	15497	0.60	7 yr
C60-C-16	LTS-31	0.70	16 months
C-32-1	15669	0.60	10 yr
C-32	15669	0.60	7 yr
C68-D-17	15669	0.70	17 months
C67-C-22	LTS-41	0.70	22 months

All pastes were prepared by a vacuum mixing procedure described elsewhere (**6**). This method permits the preparation of paste specimens containing no bubbles of entrained air, and has therefore been extremely useful in the X-ray application to be discussed presently. The fresh paste was cast either in glass or polyethylene molds. The glass molds were lined on the inner surface with paraffin wax. The specimens were cured at 25 C either in a thermostatically controlled

TABLE III.—X-RAY *d*-SPACINGS—WET SLICE AND REWETTED SAMPLE DATA FOR PASTES OF NORMAL (TYPE I) CEMENTS.

(Spacings in Å (uncorrected).)

Possible Contributing Product[a]	C-88	C-54	C-87	C-75	C-75[b]	C66-K-11	C66-O-6	C-61-C-12	C61-C-12	C61-C-17
A	...	...	...	...	...	...	9.7	9.8	9.8	9.8
B	...	8.9	...	8.9	9.0	9.0	...	9.0	9.0	9.0
K	...	...	...	...	...	...	8.7	8.6	...	...
C	...	...	...	...	...	...	...	...	8.3	...
D	7.8	7.8	7.8	7.7	7.8	7.8	7.8	7.8	7.7	7.8
E	...	...	...	...	...	...	...	7.4	7.3	7.2
A	...	...	...	...	...	...	5.66	5.68	5.63	5.60
F	5.09	5.06	5.09	5.09	[c]	5.09	5.11	5.08[d]	[c]	[c]
G	4.94	4.92	4.93	4.93	4.93	4.94	4.92	4.94	4.92	4.92
A	...	...	...	...	...	...	...	...	4.71	4.77
B	...	...	...	4.49	4.48	...	...	4.48	4.48	4.49
F	4.41	4.38	4.43	4.39	4.40	4.41	4.39	4.41	4.40	4.41
K	...	4.27	4.27	...	4.28	...	4.27	...	...	...
A, B	4.00	4.00	4.03	4.02	4.02	...	...	4.02	4.02	4.02
A, C, H	3.88	3.86	3.86	3.87	3.86	3.88	3.88	3.88	3.86	3.88
D	3.79	3.78	3.77	3.77	...	3.78	...	3.81	...	...
C	...	...	...	...	3.67	...	...	...	...	...
A	...	...	...	...	...	3.50	...	3.50	3.48	...
K	...	3.43	...	3.45	3.42	...	3.45	...	...	...
K	3.35	3.35	{3.34}	3.35	...	...	{3.34}	3.36	...	...
F	3.31	3.30		3.31	3.32	3.32		3.31	3.31	3.32
F, G	3.12	3.11	3.12	3.12	3.12	3.12	3.12	3.12	3.11	3.11
H, I	3.05	3.04	3.05	3.05	3.04	3.05	3.05	3.05	3.04	3.04
K	...	2.97	...	...	2.97	...	2.98	...	...	...
B, D	2.88	2.88	2.88	2.88	2.89	2.89	2.88	2.89	2.89	2.88
K	...	...	...	2.84	...	2.83	...	...	...	2.86
A, F, I, J	2.78	2.77	2.78	2.78	2.78	2.78	2.79	2.78	2.78	2.77
E	...	...	...	...	...	2.69	2.69	...	...	...
G	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63
A	2.60	2.58	2.59	2.59	2.58	2.59	2.59	2.59	2.56	...
D, F	2.53	2.52	2.54	2.53	2.54	...	2.54	2.54	2.53	2.53
H	2.50	2.50	2.50	2.49	...	2.49	2.49	...	2.51	2.50
B, C, G	2.47	2.45	2.46	2.47	2.46	...	2.46	2.46	2.45	2.46
B, D, F	2.43	2.42	2.43	2.43	2.42	2.43	2.43	2.43	2.43	2.43
A, D	2.37	2.37	2.36	2.36	...	2.36	2.37	2.37	2.36	2.38
K	...	2.31	2.31	...	...	...	...	...	...	...
H	2.29	2.29	2.29	2.29	2.29	2.29	2.29	2.30	2.29	...
F	2.27	2.26	2.27	2.27	2.26	2.27	2.26	2.27	2.27	2.27
A, B, J	...	...	2.19	2.19	2.20	2.19	2.20	2.20	2.20	2.21
A, C	2.14	...	...	2.14	2.14	2.14	2.14	2.14	2.15	2.13
D, H	2.10	2.10	2.10	2.10	2.10	2.11	2.10	2.10	2.11	2.10
B	2.07	2.06	2.06	...	2.07	2.07	...	2.04	2.06	2.07
D, F	2.01	2.01	2.02	2.01	2.01	2.01	2.02	2.01	2.01	2.01
A, D, F	1.96	1.96	1.97	...	1.96	1.96	1.96	1.96	...	...
G, H	1.932	1.929	1.931	1.931	1.930	1.932	1.930	1.931	1.932	1.929
H	1.912	1.912	1.912	1.912	...	...	1.912	...	...	1.912
H	1.881	1.875	1.879	1.879	1.875	...	1.879	1.881	1.877	1.883
B, I	1.826	1.819	1.824	1.824	1.823	1.827	1.823	1.828	1.826	1.828
G	1.799	1.796	1.799	1.799	1.798	1.799	1.798	1.799	1.797	1.799
F	1.722	1.714	1.723	1.719	1.717	1.719	1.723	1.723	1.719	1.723
D, G	1.690	1.688	1.689	1.690	1.688	1.690	1.688	1.690	1.690	1.689
{B, C, D, F}	{1.660}	{1.658}	1.665 1.660	{1.660}	{1.658}	{1.662}	1.662 1.652	{1.661}	1.663 1.654	1.665 1.653
B, G	1.639	1.635	1.638	1.638	1.635	1.638	1.637	1.639	1.641	1.639

---

[a] The letters in this column represent the following substances: A, ettringite; B, calcium monosulfoaluminate; C, tetracalcium aluminate hydrate; D, calcium monocarboaluminate; E, unhydrated ferrite phase; F, hydrogarnet-like phase; G, calcium hydroxide; H, calcite; I, tobermorite; J, unhydrated silicate phase; K, not known.

[b] Dried sample, rewetted in a saturated atmosphere.

[c] Badly obscured by strong peak. Spacing cannot be estimated.

[d] Shoulder on strong peak. Exact *d* spacing not certain.

TABLE IV.—X-RAY *d*-SPACINGS—WET SLICE AND REWETTED SAMPLE DATA FOR PASTES OF CEMENTS LOW IN TRICALCIUM ALUMINATE AND OF FINE CEMENTS.

(Spacings in Å (uncorrected).)

Possible Contributing Product[a]	C33-1	C33-1[b]	C65-F-22	C31-1	C60-C-16	C68-D-17	C32-1	C67-C-22
A	9.8	9.8	9.8	9.7	9.8	9.8	9.8	9.8
B	...	8.8[e]	9.1	9.0	8.9	...	...	8.9[e]
C	...	...	...	8.3	8.1	...	...	...
D	7.8	7.8	7.8	7.8	7.7	7.8	7.8	7.8
E	7.4	7.4	7.3	...	...	...	...	7.3
A	5.64	5.66	5.65	5.64	5.64	5.64	5.64	5.63
F	5.06[d]	[c]	5.09[d]	5.06[d]	5.10	...	...	5.12
A	...	...	...	...	...	5.01	...	4.99
G	4.93	4.94	4.92	4.94	4.93	4.92	4.92	4.93
A	4.72	4.72	4.72	4.72	4.70	4.71	4.72	4.72
B	...	...	...	...	...	4.50	...	...
A, F	4.39	4.40	4.42	4.41	4.41	4.41	...	4.39
K	4.26	...	...	4.26	...	...	4.29	...
A, B	4.04	...	4.00	4.02	...	...	4.02	4.02
A, C, H	3.88	3.88	3.88	3.88	3.88	3.88	3.88	3.88
D	...	...	...	3.81	...	...	...	...
A, C	3.67	3.67	3.66	...	...	...	3.69	...
A	...	...	3.59	3.59	3.59	3.62	...	3.62
A	3.48	3.48	3.49	3.49	...	3.50	3.49	3.48
K	3.35	3.38	3.39	3.35	...	...	3.36	...
F	...	3.32	3.29	3.31	3.32	...	...	3.34
A	...	...	...	...	...	...	3.26	...
F, G	3.12	3.12	3.11	3.12	3.11	3.11	3.12	3.11
H, I	3.05	3.05	3.04	3.05	3.04	3.04	3.05	3.04
K	...	...	2.99	...	...	...	...	3.00
B, D	...	...	...	2.89	2.88	...	...	...
K	...	...	...	...	...	...	2.84	2.83
A, F, I, J	2.78	2.78	2.79	2.77	2.78	2.79	2.78	2.78
J	...	...	...	...	...	2.76	...	...
E	2.67	2.69	2.68	2.70	...	...	...	2.68
G	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63
A	2.57	2.58	2.57	2.56	2.58	2.57	2.56	2.58
D, F	2.54	2.53	2.54	2.53	...	2.53	...	2.54
H	2.51	...	2.50	2.50	2.50	2.51	2.51	2.51
B, C, G	2.46	...	...	2.45	...	2.45	2.45	2.45
B, D, F	2.42	2.44	2.44	2.43	2.44	2.41	2.41	2.42
A, D	...	2.35	2.36	2.36	2.35	...	...	2.37
H	2.29	2.29	2.29	2.29	2.30	2.29	2.29	2.31
F	2.27	2.27	2.27	2.26	2.27	2.27	...	2.27
A, B, J	2.21	2.21	2.21	2.21	2.22	2.21	2.21	2.21
A, C	2.15	2.16	2.15	2.14	...	2.16	2.16	2.15
D, H	2.09	2.10	2.10	2.10	2.10	2.10	2.10	2.11
B	2.06	2.06	2.06	2.06	...	2.06	2.06	2.06
D, F	2.01	2.01	2.01	2.01	2.02	2.03	2.01	2.02
A, D, F	...	...	...	1.96	1.96	1.99	1.97	...
G, H	1.930	1.931	1.930	1.930	1.930	1.928	1.932	1.932
H	1.912	...	1.912	1.912	1.910	1.916	1.912	...
H	1.879	...	1.883	1.875	1.882	1.880	1.879	...
B, I	1.823	1.824	1.825	1.823	1.826	1.825	1.826	1.823
G	1.797	1.798	1.799	1.797	1.799	1.799	1.801	1.799
F	1.722	1.720	1.726	1.717	1.720	1.729	...	1.725
D, G	1.689	1.689	1.690	1.688	1.689	1.689	1.689	1.690
{B, C, D, F}	{1.657}	1.663 1.656	{1.660}	1.667 1.654	{1.660}	{1.664}	{1.665}	1.668 1.662
B, G	1.636	1.638	...	1.638	1.641	1.637	1.634	1.640

---

[a] The letters in this column represent the following substances: A, ettringite; B, calcium monosulfoaluminate; C, tetracalcium aluminate hydrate; D, calcium monocarboaluminate; E, unhydrated ferrite phase; F, hydrogarnet-like phase; G, calcium hydroxide; H, calcite; I, tobermorite; J, unhydrated silicate phase; K, not known.

[b] Dried sample, rewetted in a saturated atmosphere.

[c] Badly obscured by strong peak. Spacing cannot be estimated.

[d] Shoulder on strong peak. Exact *d* spacing not certain.

[e] Line is very weak. Its presence is questionable.

water bath or in a controlled temperature room. The general characteristics of the pastes are given in Table II.

Upon removal of the paste cylinders from the molds, the portion of the paste to be dried was transferred to a gloved box containing carbon dioxide-free air. The specimen was then dried for several days in the vacuum drying apparatus described elsewhere (7). The material was then ground to pass a U. S. Standard No. 200 sieve, the grinding operation being performed in a carbon dioxide-free atmosphere. The specimen was then dried to equilibrium with the vapor pressure of ice at −78 C ($\frac{1}{2}$-μ pressure)

TABLE V.—X-RAY *d*-SPACINGS—HYDRATED CEMENTS - DRIED POWDER DATA.

Possible Contributing Product[a]	C-75	C-49	C-61-C-12	C21-1W1	C-33-1	C-33	C65-F-13	C-31-1	C-31	C-32-1	C-32
B	8.2	8.2	8.2	8.4[d]	...	...	8.2	8.2	8.2	...	...
C, D	7.7	7.9	7.7	7.7	7.8	7.8	7.8	7.7	...	...	...
E	...	...	7.3	7.3	7.4	7.3	7.3	...	...	...	...
K	5.47	5.48	5.45	...	5.47	5.43	5.47	5.45	...	5.47	5.48
F	5.06[c]	[b]	[b]	5.07	5.06[c]	5.06	[b]	5.07	5.06[c]	[b]	[b]
G	4.92	4.95	4.91	4.92	4.92	4.92	4.93	4.92	4.92	4.92	4.92
F	4.41	4.41	4.41	4.38	4.41	4.39	4.38	4.39	4.39	4.40	4.39
B	4.09	4.10	4.07	...	...	...	4.08	4.08	4.08	...	...
K	...	...	...	...	...	4.00	...	...	...	...	...
C, H	3.89	3.92	...	3.86	3.86	3.89	3.85	3.86	...	...	3.92
C	3.62	...	...	...	...	3.66	3.67	...	...	...	...
F	3.31	3.32	3.31	3.30	3.32	3.35	3.31	3.31	3.30	...	3.30
	...	...	...	3.19	...	...	...	...	3.21	...	3.18
F, G	3.12	3.12	3.12	3.10	3.12	3.12	3.11	3.12	3.12	3.12	3.11
H, I	3.04	3.05	3.03	3.03	3.05	3.04	3.03	3.04	3.05	3.03	3.03
	2.92	...	...	...	...	...	2.93	2.92	...	...	...
B, C, D	2.88	2.88	2.88	2.88	...	...	...	2.86	2.86	...	2.87
B, F, I	2.78	2.78	2.78	2.78	2.78	2.77	2.78	2.77	2.76	2.79	2.78
B, C, E	...	2.70	2.71	2.68	2.67[c]	2.68	2.71	...	...	...	...
G	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63	2.63
K	...	2.60[c]	2.59	2.58	2.60	2.58	2.58	2.55	2.58	...	...
F	2.53	2.53	2.53	2.53	2.53	2.53	2.53	2.53	2.53	2.53	...
B, H	2.49	...	2.48	...	...	...	...	...	...	...	...
B, C, G	2.46	2.46	...	2.45	2.46	2.45	2.46	2.46	2.47	...	2.45
B, D, F	2.43	2.43	2.43	2.43	2.44	...	...	2.43	2.42	...	...
B	2.35	2.35	2.35	...	...	...	...	2.35	2.35	...	...
K	2.31	2.30	...	2.30	2.30	...	...	...	2.30	2.30	...
F	2.26	2.26	2.26	2.27	2.26	2.26	2.28	2.26	2.28	...	...
B, C, H	2.08	2.10	...	2.10	...	...	2.11	...	2.08	...	...
C	...	2.05	...	2.06	2.06	2.05	2.06	...	...	...	2.05
F	2.01	2.01	2.01	2.01	2.01	2.01	1.99	2.01	2.00	2.01	2.02
G, H	1.93	1.93	1.93	1.93	1.93	1.93	1.93	1.93	1.93	1.93	1.93
H	1.88	1.89	...	...	...	...	1.88	...	...	...	1.89
D, I	1.82	1.82	1.83	1.82	1.82	1.82	1.82	1.82	1.82	1.82	1.82
F, G	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80	1.80
F	1.72	1.72	1.72	1.72	1.72	1.72	1.71	1.72	1.71	...	1.71
G	1.69	1.69	1.69	1.69	1.69	1.69	1.69	1.69	1.69	1.69	1.69
B, C, D, F	1.65	1.65	1.65	1.65	1.65	1.65	1.65	1.65	1.65	1.65	1.66
G	1.64	1.64	1.64	...	1.64	1.64	1.64	1.64	1.64	...	1.64

---

[a] The letters in this column represent the following substances: A, ettringite; B, calcium monosulfoaluminate; C, tetracalcium aluminate hydrate; D, calcium monocarboaluminate; E, unhydrated ferrite phase; F, hydrogarnet-like phase; G, calcium hydroxide; H, calcite; I, tobermorite; J, unhydrated silicate phase; K, not known.

[b] Badly obscured by strong peak. Spacing cannot be estimated.

[c] Shoulder on strong peak. Exact *d*-spacing not certain.

[d] Line is very weak. Its presence is questionable.

in the vacuum drying apparatus. The dried material was stored in paraffin-sealed weighing bottles until used.

Samples were brought to equilibrium with various relative water vapor pressures over the appropriate saturated salt solutions (8) in evacuated desiccators. The samples were maintained thus for two weeks, after which they were stored in paraffin-sealed weighing bottles until used.

X-ray Apparatus:

The X-ray apparatus was a North American Philips Geiger counter diffractometer. Radiation used was Cu K_α. All specimens were scanned at a rate of $\frac{1}{8}$ deg 2θ per min. The sample chamber was sealed by a strip of polyethylene so as to reduce carbonation, as was described elsewhere (9).

Preparation of Diffractometer Specimens:

After a paste cylinder had been removed from its mold and after drying, a slice 3.5 cm long, 2.5 cm wide and about 1 mm thick was removed from the cylinder by means of a small diamond saw. The slice was taken along the axis and usually from the center portion of the cylinder. The surface of the slice was then polished smooth first with fine carborundum on a steel lap, then with white rouge on a lead lap. The specimen was kept saturated during the entire operation. The slice was mounted in the diffractometer in the same way as is a powder specimen holder.

Powder specimens were packed in conventional Philips sample holders, precautions being taken to insure that the sample had a smooth surface.

RESULTS AND DISCUSSION

The Calcium Silicate Hydrate Phase:

It has been shown that the calcium silicate hydrate produced by the hydration of tricalcium silicate or β-dicalcium silicate is a member of the tobermorite series (10,11). The X-ray diffraction pattern of this substance is made up of lines representing spacings at 3.05, 2.79, and 1.82 A.

The three lines characteristic of this calcium silicate hydrate are observed also in X-ray diffraction patterns of portland-cement paste and the appearance of the lines is similar. Lines ascribable to tobermorite were observed by Taylor in patterns obtained from cement pastes by the Debye-Scherrer photographic technique (12) and by the present authors in patterns from wet or dried hydrated cement pastes by the diffractometer technique (Tables III, IV and V). The line at 3.05 Å is a broad hump, quite asymmetric, falling off relatively sharply on the low angle side and much more gradually on the high angle side. In the patterns of the wet slices, all of which were somewhat carbonated, the 3.03 Å line of calcite is superimposed on the broad line of the silicate, at 3.05 Å.

The 2.78 Å line produced by the pastes is stronger than the 2.79 Å line produced by pastes of hydrated calcium silicate; moreover, the shape is that expected if a line from a well-crystallized hydrate were superimposed upon the 2.79 Å tobermorite line.

The 1.82 Å tobermorite line is readily observable; no known hydration product of portland cement, other than tobermorite, produces a line of this spacing of sufficient intensity to account for the line observed.

Thus the results show that the calcium silicate hydrate lines appearing in patterns of hydrated pastes of tricalcium silicate and β-dicalcium silicate are like those found in patterns of hydrated portland-cement pastes, indicating, although since only three lines are found in both cases, that the tobermorites formed in the two cases are similar.

The Calcium Sulfoaluminates:

X-ray diffraction lines of both types of sulfoaluminate, high sulfate form and low sulfate form, have been observed in hydrated portland-cement pastes. In some pastes, either one or the other of these sulfoaluminates may be detected while in others both appear simultaneously.

The two strongest ettringite (high-sulfate sulfoaluminate) ($Ca_3Al_2O_6 \cdot 3CaSO_4 \cdot 31H_2O$) lines, at 9.73 and 5.61 Å (13) occur in positions not interfered with by other possible products of cement hydration. It can be seen in Tables III and IV that whenever the 9.73 Å line appears the 5.61 Å line also appears. In some pastes, these lines are weak so that the other weaker ettringite lines are obscured by normal background variation; in others the stronger ettringite lines are more intense and some of the weaker lines can also be distinguished.

In carbonated samples the rather strong ettringite line occurring at 3.88 Å is somewhat obscured by a calcite line. However, as this calcite line is weak relative to the principal calcite line, at 3.03 Å, the 3.88 Å line found in all cement paste patterns showing the 9.73 and 5.61 Å lines is easily seen to be too strong to be accounted for only as calcite.

As can be seen from Tables III and IV, the ettringite lines are found in some pastes which have been cured for long periods of time. Thus, the data for ten-year old pastes of cements 15622, 15497, and 15669 indicate the presence of ettringite, although the weak lines in the 15497 paste pattern indicate that not much of the material is present.

The ettringite lines in pastes of cement 15754 and LTS-17 appear to decrease in intensity with time. Thus, the ettringite lines found in pastes of cement LTS-17 are less intense at 17 months than at 12 months. No such lines are found in pastes of cement 15754, except in one cured only 6 months. It appears that this cement behaves in the manner described by Taylor (12), that is, ettringite forms and then disapppears. On the other hand, the persistence of ettringite in pastes of some cements is in agreement with the observations of Turriziani (14).

The principal diffraction peaks for the low-sulfate sulfoaluminate ($Ca_3Al_2O_6 \cdot CaSO_4 \cdot 12H_2O$) are at 8.9 and 4.45 Å (15,16). The 8.9 Å line has been observed in some of the wet slice patterns (Tables III and IV). Whenever a line at 4.45 Å is present, that at 8.9 is also present. In some instances this 4.45 line is not resolved from the line of another substance at 4.40 Å. The other lines of calcium monosulfoaluminate are either too weak to be detected or are obscured by lines of other substances.

The low-sulfate sulfoaluminate lines do not behave in the same way as those of ettringite when the material is dried. While the lines of ettringite disappear on drying the basal reflections of the low-sulfate sulfoaluminate shift to lower *d*-spacings, although its other lines show few significant changes. The authors have found that when the pure material is dried in carbon dioxide-free air at 115 C or *in vacuo* at room temperature to the 0.5-μ level the 8.9 Å line, assigned 001 by Fratini, Schippa, and Turriziani (16), contracts to 8.2 Å, and the line at 4.45 Å, assigned 002, contracts to 4.1 Å in agreement with Roberts' observations (17). The 8.2 Å line appears in the patterns of those pastes for which the wet slab pattern showed the 8.9 Å line. In the past, the 8.2 Å line has been attributed to tetracalcium aluminate hydrate (12), but since it appears only in patterns of dry material, the wet counterpart of which shows the low-sulfate sulfoaluminate line, it is apparent that it is the latter substance which is responsible for the line. Likewise, the 4.15

Å line in dried powder patterns appears only when the 8.2 Å line does.

The lines of low-sulfate sulfoaluminate appearing in dried powder patterns of well-hydrated pastes are strong and sharp, indicating that a significant amount of the material is present in the paste.

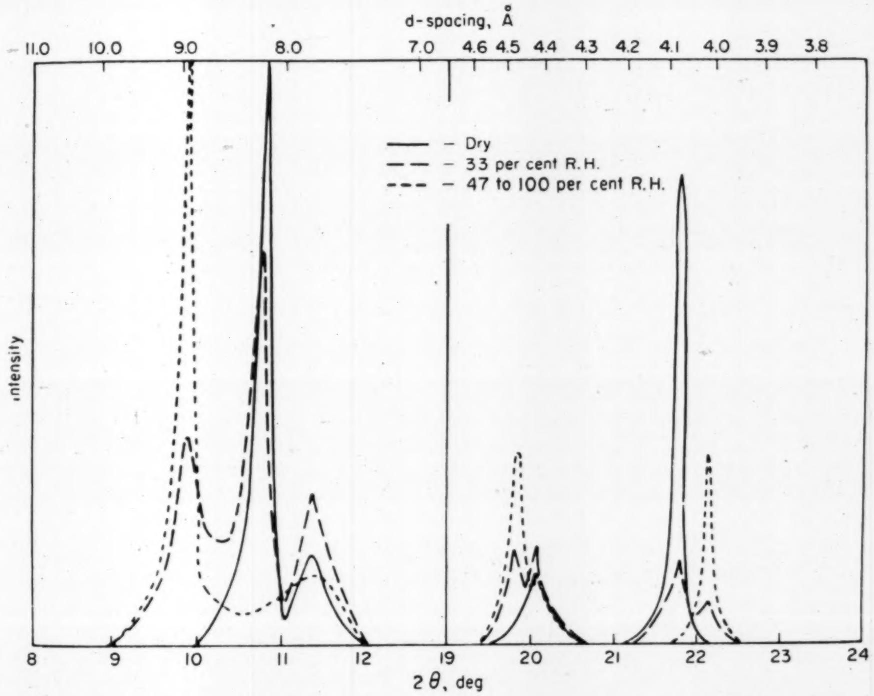


FIG. 1.—Calcium Monosulfoaluminate Lines in X-ray Patterns of Portland-Cement Paste at Different Relative Humidities.

Patterns were examined of a group of pastes of cement 15754 hydrated from 8 hr to 14 days, none of which was completely hydrated. The low-sulfate sulfoaluminate lines were absent in the pattern of the 8-hr sample but were observable in patterns of specimens 1 day old or older. The lines appeared fairly sharp, and their intensity increased with specimen age. As these specimens had been prepared for another purpose, wet slice data were not obtained; hence the presence of ettringite in them was not established by direct observation. However, the absence of both low-sulfate sulfoaluminate and gypsum lines in the youngest specimen and the growth of the low-sulfate sulfoaluminate lines with time is a further indication that ettringite is the first sulfoaluminate to form and that it then converts to the monosulfoaluminate.

Dried specimens of several of the pastes listed in Tables I and II were brought to equilibrium with various relative humidities. No significant changes were observed at humidities below 28 per cent. However, in patterns of low-sulfate sulfoaluminate-bearing samples equilibrated at higher humidities, the 8.9 Å line appeared. It showed only as a shoulder on the 8.2 Å peak at

28.8 per cent relative humidity, was about equal in intensity with the shrinking 8.2 Å peak at 33 per cent relative humidity, and at 42 per cent relative humidity, the 8.2 Å peak was a small shoulder on the 8.9 Å peak. These features are illustrated in Fig. 1. Also shown in Fig. 1 is the 7.8 Å calcium monocarboaluminate line, to be discussed below. At higher humidities, the patterns appeared quite like the wet slice patterns when the effect of partial carbonation of the wet slabs is considered. The low-sulfate sulfoaluminate lines in the rewetted samples had intensities comparable to those of the lines in the dried powder patterns. That is, they were strong and sharp. Because of this, it was easy to distinguish the 4.45 Å sulfoaluminate line from a 4.40 Å line due to another phase, to be discussed below. This resolution is illustrated in Fig. 1. Furthermore, it can be seen from the results in Tables III, IV, and V and Fig. 1 that when the 4.1 Å line in the dry material shifts to 4.45 Å in the wet material, the line at 4.0 Å reappears as well. This latter corresponds to the line reported at 3.99 Å by Midgely (15) and at 4.02 Å by Fratini, Schippa, and Turriziani (16).

Thus, in Fig. 1, idealized patterns in the 9 Å and 4 Å regions are shown for pastes containing the dry form of low-sulfate sulfoaluminate, the wet form, and an intermediate case (33 per cent relative humidity) in which both forms appear simultaneously. The differences between wet slab and rewetted powder patterns shows that the low-sulfate sulfoaluminate reacts with carbon dioxide just as does calcium hydroxide.

Rewetting pastes containing ettringite at relative humidities up to 47 per cent had no significant effect on the diffraction pattern, but upon rewetting at 100 per cent relative humidity, the ettringite lines return. No significant amount of carbonation occurred with these rewetted samples, and yet the ettringite lines were of comparable intensities to those in the original wet slice patterns wherein considerably more carbonation had occurred. Thus, if carbon dioxide reacts with ettringite, it is sufficiently slow so as not to affect the X-ray observations.

The low-sulfate sulfoaluminate has been found in pastes of the high tricalcium aluminate cements (1,2) investigated, but in pastes of only one of the low tricalcium aluminate cements examined, namely, LTS-25. On the other hand, ettringite is found in pastes of cements 15754 and LTS-17 but as was pointed out above only at early ages. It was observed in pastes of all the other cements, but in small amounts in pastes of the cements 15497 and LTS-31 which contained an apparently greater amount of low-sulfate sulfoaluminate, based on intensity considerations. Ettringite was the *only* sulfoaluminate found in pastes of the cements 15669 and LTS-41.

Pastes of cement 15669 are of especial interest in one respect. If the low-sulfate sulfoaluminate were to have formed instead of ettringite, not enough alumina would be present in the cement to react with all the SO_3. Under these circumstances, it is possible that a sulfoferrite might form. Malquori and Cirilli (18) have prepared calcium sulfoferrites analogous to the calcium sulfoaluminates found in hydrated cement pastes. The X-ray diffraction patterns of these sulfoferrites are, in general, very much like those of their sulfoaluminate analogs. As a result, the sulfoferrite compounds, were they present in portland-cement pastes, would not be readily distinguishable from their aluminate counterparts. However, there is evidence that the iron-bearing phase in cement hydrates in a unique manner not in-

TABLE VI.—*d*-SPACINGS REPORTED FOR TRICALCIUM ALUMINATE AND FERRITE HYDRATES AND CORRESPONDING SPACINGS IN SOME REPRESENTATIVE HYDRATED CEMENT PASTES.

hkl	$3 CaO \cdot Al_2O_3 \cdot 6H_2O$ (Reference 20)	$3 CaO \cdot Al_2O_3 \cdot 6 H_2O$ (Reference 15)	$3 CaO \cdot Al_2O_3 \cdot 6 H_2O$ (Reference 19)	$3 CaO \cdot Al_2O_3 \cdot 3.75 H_2O$ (Reference 21)	$3 CaO \cdot Fe_2O_3 \cdot 6 H_2O$ (Reference 20)	$3 CaO \cdot Fe_2O_3 \cdot 6 H_2O$ (Reference 19)	$3 CaO \cdot Fe_2O_3 \cdot 2 H_2O$ (Reference 19)	Calculated for a_o = 12.4 Å	C-75	C33-1	C21-1W1
1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*
211	5.16 (VS)	5.14 (S)	5.13 (MS)	5.03 (VW)	5.20 (M)	5.18 (MS)	5.07 (M)	5.063	5.09	5.06	5.07
220	4.47 (M)	4.45 (M)	4.45 (M)	4.39 (VW)	4.54 (S)	4.50 (S)	4.38 (MW)	4.385	4.39	4.39	4.38
321	3.37 (M)	3.37 (M)	3.36 (MW)	3.30 (VW)	3.40 (W)	3.41 (MW)	3.32 (W)	3.314	3.31	3.32	3.30
400	3.15 (M)	3.15 (M)	3.15 (W)	3.09 (VW)	3.19 (S)	3.19 (MS)	3.10 (MS)	3.100	[a]	[a]	3.10
420	2.81 (M)	2.82 (MS)	2.81 (MS)	2.77 (VW)	2.85 (S)	2.85 (MS)	2.78 (MS)	2.773	[a]	[a]	[a]
332	...	...	2.68 (VW)	...	2.72 (VW)	2.72 (VW)	...	2.644	...	...	...
422	2.56 (VW)	2.57 (W)	2.55 (W)	...	2.60 (M)	2.61 (MW)	2.53 (M)	2.531	2.53	2.53	2.53
431, 510	2.46 (W)	2.47 (MW)	2.47 (W)	...	2.50 (VW)	2.51 (W)	2.44 (VW)	2.432	2.43	2.44	2.43
521	2.30 (VS)	2.30 (VS)	2.30 (MS)	2.26 (VW)	2.33 (M)	2.33 (MS)	2.27 (W)	2.264	2.27	2.27	2.27
440	2.22 (VW)	2.23 (VW)	2.23 (W)	...	...	2.26 (W)	...	2.192	...	...	...
611, 532	2.04 (S)	2.04 (S)	2.04 (S)	2.01 (VW)	2.07 (M)	2.07 (S)	2.01 (S)	2.012	2.01	2.01	2.01
620	1.99 (VW)	1.99 (VW)	1.99 (VW)	...	1.99 (W)	2.02 (W)	1.96 (W)	1.961	1.96	...	...
444	1.81 (VW)	1.82 (VW)	1.82 (VW)	...	...	1.84 (VW)	1.79 (VW)	1.790	[a]	[a]	[a]
640	1.74 (W)	1.75 (MW)	1.75 (MS)	1.72 (VW)	1.77 (M)	1.77 (M)	1.72 (M)	1.720	1.72	1.72	1.72
633, 552, 721	1.71 (W)	1.71 (W)	1.71 (W)	...	1.73 (W)	1.74 (VW)	1.70 (VW)	1.687	[a]	[a]	[a]
642	1.68 (M)	1.68 (M)	1.68 (S)	1.66 (VW)	1.70 (VS)	1.71 (VS)	1.66 (VS)	1.657	1.66	1.66	1.65
651, 732	1.60 (VW)	1.60 (M)	...	...	1.62 (VW)	...	...	1.575	...	...	...
800	1.57 (W)	1.57 (VW)	1.57 (W)	...	1.59 (W)	1.60 (W)	1.55 (W)	1.550	...	...	...
840	1.40 (W)	1.41 (MS)	...	...	1.43 (M)	...	...	1.386	...	...	...

[a] Probably present, but overlapped by line from some other component.

volving SO_3. This will be discussed below.

The Calcium Aluminate and Calcium Ferrite Hydrates:

It can be seen from the data in Tables III, IV, and V that there is a group of lines not accounted for by tobermorite, calcium hydroxide, or one of the sulfoaluminates, which appears distinctly in patterns of most pastes. Although these lines are not clearly discernible in patterns of cements 15669 and LTS-41 (high dicalcium silicate cements) there is some evidence that they are present. The lines of this group are indicated in column 1 of Tables III, IV, and V by the letter "F." Two representative sets of these lines are listed in Table VI, columns 10 and 11. The results given in column 12 are from a water extracted paste, to be discussed below. It can be seen from Table VI that this series of lines is similar to that of tricalcium aluminate hexahydrate, $3CaO \cdot Al_2O_3 \cdot 6H_2O$, (15,19,20) but occur at smaller spacings. The series is also similar to that of the corresponding ferrite, $3CaO \cdot Fe_2O_3 \cdot 6H_2O$ (19,20) but the shift is even greater. Thus the spacings observed could not be accounted for by a solid solution of the two hexahydrates.

Burdese and Gallo (19) report spacings for a partially dehydrated cubic tricalcium ferrite hydrate, $3CaO \cdot Fe_2O_3 \cdot 2H_2O$ (see Table VI, column 8), these spacings being in very good agreement with those observed in cement paste patterns (Table VI, columns 10, 11 and 12). The lines at 3.10 and 2.78 Å, of course, are overlapped by strong lines of $Ca(OH)_2$ and the calcium silicate hydrate, respectively. The compound $3CaO \cdot Fe_2O_3 \cdot 2H_2O$, however, is obtained by dehydration of the corresponding hexahydrate at 250 C. Likewise, thermal dehydration of tricalcium aluminate hexahydrate causes a decrease in the spacings of that material. Köberich (21) reports spacings for a partially dehydrated calcium aluminate hydrate of the composition $3CaO \cdot Al_2O_3 \cdot 3.75H_2O$ (Table VI, column 5), also in good agreement with those observed in cement paste patterns.

The cubic lattice parameters reported for $3CaO \cdot Fe_2O_3 \cdot 2H_2O$ (19) and $3CaO \cdot Al_2O_3 \cdot 3.75H_2O$ (21) are 12.39 and 12.37 Å, respectively. The spacings calculated for a cubic lattice parameter of 12.40 Å corresponding to those observed in cement pastes are given in Table VI, column 9. These, too, are seen to be in excellent agreement with the spacings observed in cement paste patterns. It would be expected that if a solid solution of these two materials could form in the hydration of a cement, it would have the same *d* spacings. If the substance giving these spacings had, on the average, an Al_2O_3/Fe_2O_3 ratio of unity, as does the original ferrite phase (1,2) then the over-all composition of the phase would be approximately $6CaO \cdot Al_2O_3 \cdot Fe_2O_3 \cdot 6H_2O$. It is difficult to imagine that species normally formed by dehydration at relatively high temperatures should form at room temperature in a saturated aqueous system unless some other substance were dissolved in it to stabilize it. Stabilization of solid phases under conditions far removed from equilibrium is not an unusual phenomenon, especially in cement chemistry; one merely need consider the case of β-Ca_2SiO_4. However, the more usual examples, such as β-Ca_2SiO_4, do not involve an aqueous solution phase.

Probably the most likely substance to dissolve in a tricalcium aluminate hydrate - tricalcium ferrite hydrate phase is silica. As was shown by Flint, McMurdie, and Wells (20), the compounds $3CaO \cdot Al_2O_3 \cdot 6H_2O$, $3CaO \cdot Fe_2O_3 \cdot 6H_2O$, $3CaO \cdot Al_2O_3 \cdot 3SiO_2$ and $3CaO \cdot Fe_2O_3 \cdot 3SiO_2$ may all enter into solid solution with one another, these compounds and their solid solutions comprising the hydrogarnet system. The effect of a mole of silica in the system is to replace two moles of water. Flint, McMurdie, and Wells show that complete replacement of Fe_2O_3 by Al_2O_3 in the hydrated end member causes a contraction in lattice parameter from 12.72 to 12.54 Å, or 0.18 Å, and complete replacement of water by silica causes a lattice parameter contraction of 0.72Å. Based on their data, an equimolar solid solution of tricalcium aluminate hexahydrate and tricalcium ferrite hexahydrate would have a lattice parameter of 12.63 Å, and such a solid solution having a lattice parameter of 12.40 Å would contain 0.96 moles of silica in place of 1.92 moles of water out of 6 moles. The over-all composition of such a phase would be $6CaO \cdot Al_2O_3 \cdot Fe_2O_3 \cdot 1.92SiO_2 \cdot 8.16H_2O$. That this is actually the composition of the phase in question has not yet been determined. It requires the substitution of a considerably greater amount of silica than would be expected on the basis of considerations such as bound water content (22).

The lines ascribed to the proposed solid solution phase appear in the same positions in wet slice as well as dried powder patterns (Tables III, IV and V). In almost all patterns, the 5.07 Å line is partly obscured by the 4.90 Å calcium hydroxide line, although in many cases a shoulder appears sufficiently distinctly so as to permit a reasonable estimate of the *d* spacing. Occasionally the shoulder is not so distinct, and about all that can be said for such cases is that the line is present.

The X-ray pattern of the phase in question found in a specimen of a well-hydrated Type I (15754) paste, C22-1W1, from which most of the calcium hydroxide had been extracted with water was somewhat more distinct (Table IV, column 5 and Table VI, column 12). The 5.0 Å peak was almost completely undisturbed by the reduced 4.90 Å calcium hydroxide line. The 3.11 Å line was too large to be accounted for by the remaining calcium hydroxide, indicating the presence of the heretofore undetectable 3.12 Å line (Table VI). Although little evidence for other substances containing iron and alumina was found in this particular specimen, the 2.78 Å peak observed was still too large to be caused only by the calcium silicate hydrate phase, indicating that the tricalcium aluminate hydrate-tricalcium ferrite hydrate phase contributed to this peak, as was to be expected.

In wet slab patterns, a contribution to the 4.4 Å line is made by the low-sulfate sulfoaluminate in many cases. However, upon drying of such samples, the 4.4 Å line still appears, while a 4.1 Å line is also found. In rewetted sample patterns, wherein a good part of the low-sulfate sulfoaluminate has not been destroyed by carbonation, two distinct peaks may be observed in the 4.4 Å region. These are at 4.48 and 4.40 Å representing the low-sulfate sulfoaluminate and hydrated aluminoferrite solid solution, respectively. Of course, only one peak is observed in rewetted sample patterns of material which contains no low-sulfate sulfoaluminate.

The lines of the aluminoferrite hydrate begin to appear during the first week of hydration. Their growth may be correlated with a decrease in the 7.3 Å unhydrated ferrite line. There appears to be no significant change in appearance of the hydrated aluminoferrite lines after the disappearance of the 7.3 Å line. Hence it appears that this phase is a primary hydration product of the aluminoferrite in the original cement.

There is also X-ray diffraction evidence for the presence of other hydrates containing aluminum and possibly iron. All of the wet slice patterns contain a line at 7.8 Å, corresponding to the principal line of calcium monocarboaluminate (23). Much of this carboaluminate resulted from carbonation of the wet slices during handling. According to Schippa and Turriziani (24), it is probably this substance which was mistaken for a hexagonal tricalcium aluminate hydrate by earlier investigators.

The 7.8 Å line in wet slice patterns has a pronounced asymmetry on the low angle side in many cases. Sometimes this asymmetry is actually a shoulder, representing a partially obscured peak at about 8.3 Å. This corresponds closely to the strongest line of tetracalcium aluminate hydrate, $4CaO \cdot Al_2O_3 \cdot 13H_2O$, reported as 8.4 Å by Schippa and Turriziani (24) and as 8.2 Å by Roberts (17). In the wet slice patterns, this line is sufficiently weak that none of the other lines of this substance is detectable.

The principal peak of the iron analog of this aluminate, that is, $4CaO \cdot Fe_2O_3 \cdot 13H_2O$, occurs at 8.2 Å (25). Thus, the line observed in this region could represent a solid solution of this material with the corresponding aluminate. However, the determination of *d* spacing from a shoulder of the type described is far too inaccurate to warrant any such conclusion.

The 7.8 Å carboaluminate line is weaker in dried powder patterns because of very limited carbonation, but it still appears at 7.8 Å, as there is no shift on drying, just as none is observed upon wetting or drying a specimen of pure calcium monocarboaluminate. The spacings observed in both wet and dry specimens of the latter were in good agreement with those reported by Turriziani and Schippa (23) and by Buttler, Glasser, and Taylor (26). The 7.8 Å peaks in rewetted cement paste patterns, too, are weak, indicating that not much of the carboaluminate is present. That little, if any, carbon dioxide has entered the dried or rewetted powder is also indicated by the fact that the principal calcite line is not observable.

The 8.2 Å line of tetracalcium aluminate hydrate shifts to lower values as the material is dried, as was also shown by Buttler, Glasser, and Taylor (26). The shift to 7.8 Å is accomplished rather quickly and a further shift to 7.4 Å relatively slowly, and with only a small amount of additional dehydration. In the time it took to obtain an X-ray diffraction pattern of the 7.4 Å material, the 7.8 Å line had begun to return. A second scanning of the same specimen produced a pattern revealing no 7.4 Å line. Thus, the line observed at 7.8 Å in dried pastes may represent either carboaluminate, tetracalcium aluminate hydrate, or both.

Several investigators have reported basal spacings for tetracalcium ferrite hydrate varying from 8.2 to 7.8 Å (25,27,28). The diffraction pattern of this material so resembles that of the corresponding aluminate that it has not yet been possible to ascertain whether one or the other or both of these substances are present.

In some dried powder patterns, other lines of the tetracalcium aluminate (or ferrite) hydrate are found. The most important among these is at 3.9 Å (Table V). Lines at 2.46 and 2.10 Å also appear, but these may contain contributions from the monosulfoaluminate. The similarity of the pattern of the latter compound with that of tetracalcium aluminate hydrate is quite marked, with the exception of the locations of the basal reflections. Evidence for tetracalcium aluminate hydrate has also been found in electron diffraction investigations (29).

SUMMARY AND CONCLUSIONS

1. A group of hydrated portland cements of various types has been examined qualitatively by means of X-ray

diffraction. From the diffraction results, a number of hydration products have been identified.

2. The calcium silicate hydrate phase was found to be similar to the tobermorite formed by paste hydration of tricalcium or β-dicalcium silicate, on the basis of the number of lines observed and their spacings.

3. Both calcium sulfoaluminates have been found. In pastes of cements relatively high in tricalcium aluminate there is evidence that the high-sulfate sulfoaluminate forms first, then disappears as the low-sulfate sulfoaluminate forms. In those cements studied which are low in tricalcium aluminate, the high-sulfate form remains indefinitely, though some indication of the low form has been found. In those cements studied which are high in dicalcium silicate as well, only the high form was observed even in old specimens.

4. Evidence has been obtained for the presence of a hydrogarnet type tricalcium aluminate hydrate or a tricalcium ferrite hydrate or, more probably, a solid solution of the two. This product may contain silica too. However, the data are not precise enough to permit a good estimate of the composition of the phase.

5. Lines of calcium monocarboaluminate have been observed. These lines become stronger when the specimen carbonates. At the same time strong calcite lines appear and if the low-sulfate sulfoaluminate is present, its lines diminish in strength. The carboaluminate and calcite lines are strong in patterns of wet slices of cement paste, while the carboaluminate lines are weak and the calcite lines absent, in patterns of dried or rewetted pastes.

6. There is also present in some cases a small amount of tetracalcium aluminate hydrate. The ferrite analog may be present, or a solid solution of the two, but this cannot be ascertained from the data.

Acknowledgments:

The authors wish to express their sincere appreciation to Mr. Stephen Brunauer for his many helpful discussions and suggestions and to Mr. C. H. Weise for his assistance in preparing some of the specimens. The authors also wish to thank Mr. Nathan Greening for the samples of pure materials which he was kind enough to provide, and for helpful discussions.

REFERENCES

(1) L. E. Copeland, Stephen Brunauer, D. L. Kantro, Edith G. Schultz, and C. H. Weise, "Quantitative Determination of the Four Major Phases of Portland Cement by Combined X-ray and Chemical Analysis," *Analytical Chemistry*, Vol. 31, pp. 1521–1530 (1959). (PCA Research Dept. Bulletin No. 108.)

(2) Stephen Brunauer, L. E. Copeland, D. L. Kantro, C. H. Weise, and Edith G. Schultz, "Quantitative Determination of the Four Major Phases in Portland Cement by X-ray Analysis," *Proceedings*, Am. Soc. Testing Mats., Vol. 59, p. 1091 (1959). (PCA Research Dept. *Bulletin No. 113.*)

(3) H. H. Steinour, "The Reactions and Thermochemistry of Cement Hydration at Ordinary Temperature," *Proceedings*, Third International Symposium on the Chemistry of Cement, Cement and Concrete Association, London, 1952, pp. 261–289. (PCA Research Dept. *Bulletin No. 44.*)

(4) T. C. Powers and T. L. Brownyard, "Physical Properties of Hardened Portland Cement Paste, Part 2–Studies of Water Fixation," Journal Am. Concrete Inst., *Proceedings*, Vol. 43, pp 249–336 (1947). (PCA Research Dept. *Bulletin No. 22.*)

(5) William Lerch and C. L. Ford, "Long-Time Study of Cement Performance in Concrete. 4. Chemical and Physical Tests of the Cements," *Journal*, Am. Concrete Inst., *Proceedings*, Vol. 44, pp. 743–795 (1948). (PCA Research Dept. *Bulletin No. 26.*)

(6) T. C. Powers, L. E. Copeland, J. C. Hayes and H. M. Mann, "Permeability of Portland Cement Paste," *Journal*, Am. Concrete Inst., *Proceedings*, Vol. 51, pp. 285–298 (1955). (PCA Research Dept. *Bulletin No. 53.*)

(7) L. E. Copeland and J. C. Hayes, "The Determination of Non-Evaporable Water in Hardened Portland Cement Paste," ASTM BULLETIN, No. 194, Dec., 1953, pp. 70–74. (PCA Research Dept. *Bulletin No. 47.*)

(8) R. H. Stokes and R. A. Robinson, "Standard Solution for Humidity Control at 25° C," *Industrial and Engineering Chemistry*, Vol. 41, 2013, (1948).

(9) L. E. Copeland and R. H. Bragg, "Quantitative X-ray Diffraction Analysis," *Analytical Chemistry*, Vol. 30, pp. 196–201 (1958). (PCA Research Dept. *Bulletin No. 88.*)

(10) Stephen Brunauer, L. E. Copeland and R. H. Bragg, "The Stoichiometry of Hydration of Tricalcium Silicate at Room Temperature. II. Hydration in Paste Form," *Journal of Physical Chemistry*, Vol. 60, pp. 116–120 (1956). (PCA Research Dept. *Bulletin No. 65.*)

(11) Stephen Brunauer, D. L. Kantro and L. E. Copeland, "The Stoichiometry of the Hydration of Beta-Dicalcium Silicate and Tricalcium Silicate at Room Temperature," *Journal*, Am. Chemical Soc., Vol. 80, pp. 761–767 (1958). (PCA Research *Bulletin No. 86.*)

(12) H. F. W. Taylor, "Studies on the Hydration of Portland Cement," Paper given at 27th Congress of Industrial Chemistry, Brussels, 1954, 4 pp.

(13) H. E. Swanson, N. T. Gilfrich, M. I. Cook, R. Stinchfield and P. C. Parks, "Standard X-ray Diffraction Powder Patterns," Vol. 8, pp. 3–4, Nat. Bureau Standards, *Circular No. 539*, April, 1959.

(14) R. Turriziani, "The Hydration Processes of Portland Cement," *L'Industria Italiana del Cemento*, Vol. 29, (7, 8) pp. 185–189, (9) pp. 219–223, (10) pp. 244–246, (11) pp. 276–280 (1959).

(15) H. G. Midgley, "A Compilation of X-ray Powder Diffraction Data of Cement Minerals," *Mag. Concrete Research*, Vol. 9, pp. 17–24 (1957).

(16) N. Fratini, G. Schippa and R. Turriziani, "X-ray Analysis of the Calcium Sulfoaluminate Hydrates," *Ricerca Scientifica*, Vol. 25, pp. 57–61 (1955).

(17) M. H. Roberts, New Calcium Aluminate Hydrates," *Journal of Applied Chemistry*, Vol. 7, pp. 543–546 (1957).

(18) G. Malquori and V. Cirilli, "Hydrated Calcium Ferrites and Complexes Produced from Tricalcium Ferrite by Association with Various Calcium Salts. II," *Ricerca Scientifica*, Vol. 11, pp. 434–442 (1940).

(19) A. Burdese and S. Gallo, "Dehydration of Tricalcium Ferrite," *Annali di Chimica*, Vol. 42, pp. 349–355 (1952).

(20) E. P. Flint, H. F. McMurdie and L. S. Wells, "Hydrothermal and X-ray Studies of the Garnet-Hydrogarnet Series and the Relationship of the Series of the Hydration Products of Portland Cement," *Journal of Research*, Nat. Bureau of Standards, Vol. 26, pp. 13–33 (1941).

(21) K. Koberich, "Maximum Vapor Pressure and Adsorption Equilibrium," Doctoral Dissertation, Friedrich Wilhelm University, Berlin, 1934.

(22) Stephen Brunauer, D. L. Kantro and L. E. Copeland, Unpublished calculations.

(23) R. Turriziani and G. Schippa, "The Existance of a Hydrated Calcium Monocarboaluminate," *Ricerca Scientifica*, Vol. 26, pp. 2792–2797 (1956).

(24) G. Schippa and R. Turriziani, "Hydrated Calcium Aluminates," *Ricerca Scientifica*, Vol. 27, pp. 3654–3661 (1957).

(25) Paul Seligmann and Nathan Greening, unpublished data.

(26) F. G. Buttler, L. S. Dent Glasser and H. F. W. Taylor, "Studies on $4CaO\text{-}Al_2O_3\text{-}13H_2O$ and the Related Natural Mineral Hydrocalumite," *Journal*, Am. Ceramic Soc., Vol. 42, pp. 121–126 (1959).

(27) K. Watanabe and T. Iwai, "Electron-Microscopic and X-ray Investigation of Tetracalcium Aluminoferrite and Dicalcium Ferrite," *Semento Gijutsu Nenpo*, Vol. 9, pp. 57–68 (1955).

(28) H. F. W. Taylor, private communication.

(29) L. E. Copeland and E. G. Schulz, unpublished data.

DISCUSSION

Mrs. K. Mather.[1]—I am interested that the author was working with fairly old, well-hydrated pastes. Most of the specimens that we have examined have been either field concrete, or pastes, say, 2 to 3 yr old. In such pastes there are enough remnants of the strong peaks of the portland cement in the region from 31 to 36 deg 2-θ, where you would like to be able to see more of the lines of the hydration products, so that it has been a considerable problem to us.

Mr. D. L. Kantro (*author*).—We intentionally selected very well-hydrated specimens. As a matter of fact, the first specimens we examined were 7 to 11 yr old. Even at these ages, specimens with low water-cement ratios, such as 0.4, give small residual peaks for dicalcium silicate and sometimes for the ferrite phase. However, these peaks do not obscure the stronger peaks, especially in the 32-deg region where various important hydration product lines including one of tobermorite occur. The ferrite line at about 7.6 Å is just out of the way of the low angle region in which we are most interested. In general, clinker lines from pastes better than 90 per cent hydrated do not seriously interfere with the important hydration product lines.

---

[1] Chief, Petrography Section, Concrete Division, Waterway Experiment Station, Jackson, Miss.

INVESTIGATION OF THE FINE AND COARSE AGGREGATES OF NORTHERN ILLINOIS FOR ALKALI REACTIVITY*

By J. F. Weigel[1]

Synopsis

The alkali expansion characteristics of the fine and coarse aggregates from six major sources in northern Illinois were studied. Structures in which the suspect aggregates had been used over a period of years were inspected and six different test methods were used to evaluate reactivity. Results given include length changes of test specimens at ages up to $1\frac{1}{2}$ yr and expansion measurements of concrete in structures at ages up to 2 yr.

Inspection of existing structures and a review of service performance of aggregates failed to disclose any distress attributable to alkali reactivity. Results of expansion measurements of mortar bars, concrete prisms, and concrete in structures, made with high alkali cement and suspect aggregates, show no deleterious expansion. However, based on petrographic and chemical evaluation, several of the aggregates would be considered borderline and several, alkali reactive.

Inspection of a number of distressed concrete structures in Northern Illinois during early 1957 led several investigators to believe that cracking appearing in these structures was due to alkali expansion in the concrete. Additionally, subsequent petrographic examination of aggregate from several deposits in the Northern Illinois area led the investigators to further suspect that the local aggregates were alkali reactive and responsible for concrete distress in the area. The investigation up to this point, however, was by no means conclusive, and as the service performance of the aggregates from this area had never disclosed evidence of distress due to alkali expansion, it was felt the matter should be thoroughly investigated.

In January of 1958, The Portland Cement Assn. and The Medusa Portland Cement Co., with Herbert Insley serving as consultant, began an investigation of the alkali expansion characteristics of the suspect aggregates. To assure broad coverage, it was decided to employ all of the recognized techniques used in predicting potential alkali reactivity in aggregates. These included the following:

A field trip to inspect existing structures for alkali expansion distress;

A lithologic and petrographic examination of the fine and coarse aggregates;

Test for potential reactivity of aggregates (ASTM Method C 289);[2]

Aggregate examination, using osmotic cell flow test technique;

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Assistant Vice-President, Medusa Portland Cement Co., Cleveland Heights, Ohio.

[2] Tentative Method of Test for Potential Reactivity of Aggregates (Chemical Method) (C 289 – 57 T), 1958 Book of ASTM Standards, Part 4, p. 522.

TABLE I.—LITHOLOGIC ANALYSIS OF DIFFERENT SIZE FRACTIONS OF GRAVELS AND SANDS, IN PER CENT OF NUMBER OF PARTICLES.[a]

	Retained on 3/8 in. Sieve[b, c]	Passing No. 16 Sieve Retained on No. 30 Sieve[d]	Passing No. 30 Sieve Retained on No. 50 Sieve[d]	Passing No. 50 Sieve Retained on No. 100 Sieve[d, e]	Weighted Average
Forreston, Ill.					
Quartz	3.9	29.8	63.4	56.5	13.2
Limestone	77.7	29.4	9.4	16.8	65.8
Chert	12.7	2.0	0.2	6.0	10.6
Other	5.7	38.8	27.0	20.7	10.4
Elgin, Ill.					
Quartz	0.4	20.2	40.8	46.5	12.8
Limestone	92.0	15.9	10.2	24.0	60.9
Chert	4.3	1.7	0.6	1.3	3.0
Other	3.3	62.2	48.4	28.2	23.3
Algonquin, Ill.					
Quartz	0.6	20.4	32.9	40.1	9.8
Limestone	93.2	18.3	12.3	30.2	67.5
Chert	3.3	1.1	1.5	1.7	2.7
Other	2.9	60.2	53.3	28.0	20.0
Plainfield, Ill.					
Quartz	0.6	6.4	15.6	19.3	5.8
Limestone	96.5	42.7	32.0	31.6	67.0
Chert	1.6	1.4	0	1.1	1.2
Other	1.3	49.5	52.4	48.0	26.0
South Beloit, Ill.					
Quartz	8.1	33.0	64.8	57.9	28.8
Limestone	73.5	26.7	12.2	15.2	48.0
Chert	9.0	3.0	0.3	1.8	5.5
Other	9.4	37.3	22.7	25.1	17.7
Rockford, Ill.					
Quartz	15.2	35.1	51.6	50.3	34.5
Limestone	62.4	16.3	10.0	16.7	32.1
Chert	11.2	2.5	0.7	1.3	5.2
Other	11.2	46.1	37.7	31.7	28.2

[a] The Jones riffle sampler was not used because of the small number of particles in each sieve size. Instead, the usual method of sampling with mixing and quartering was used. For the sieve sizes larger than 3/8 in., the total number of particles in each fraction were counted.

[b] The size fraction 3/8 in. to No. 4 was reserved for gravity separations and was not available for this lithologic analysis.

[c] Size fractions No. 4 to No. 8 and No. 8 to No. 16 were omitted from this analysis to save time.

[d] The size fractions No. 16 to No. 30 and No. 30 to No. 50 were counted with the low power microscope and the size fraction No. 50 to No. 100 in a 1.540 refractive index immersion liquid with the petrographic microscope.

[e] The "pan" fraction (all through No. 100 sieve) was omitted from the analysis due to the large amounts of fine dust included and the consequent inaccuracy of the count. Petrographic inspection of the "pan" fractions did not indicate marked differences from the No. 50 to No. 100 fractions.

Expansion measurement of prisms of the limestone fraction of the coarse aggregate after submersion in sodium hydroxide solution;

Expansion of mortar bars (ASTM Method C 227);[3]

Expansion of concrete prisms; and

---

[3] Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 – 58 T), 1958 Book of ASTM Standards, Part 4, p. 514.

Expansion measurements in concrete structures in which the suspect aggregates had been used.

Seven of the aggregate-producing locations in the area were inspected by L. S. Brown and Herbert Insley, and arrangements were made to obtain 500-lb samples of the fine and coarse aggregates from six of the aggregate producers. The locations of those producers offering aggregate samples were: Forreston, Ill., Elgin, Ill., Algonquin, Ill., Plainfield, Ill., South Beloit, Ill., and Rockford, Ill.

The aggregate samples were prepared for tests at the laboratories of the Portland Cement Assn., and a 20-lb sample representative of each lot was forwarded to Herbert Insley for his examination.

TABLE II.—PERCENTAGE OF TOTAL CHERT (BY PARTICLE SIZE) IN ALL SIZE FRACTIONS OF ILLINOIS GRAVELS.

	Retained on 3/8 in. Sieve	Passing 3/8 in. Sieve Retained on No. 4 Sieve	Passing No. 8 Sieve Retained on No. 16 Sieve	Passing No. 16 Sieve Retained on No. 30 Sieve	Passing No. 30 Sieve Retained on No. 50 Sieve	Passing No. 50 Sieve Retained on No. 100 Sieve	Weighted Average
Forreston, Ill.	12.7	15.8	7.1	2.0	0.2	6.0	11.5
Elgin, Ill.	4.3	4.8	6.3	1.7	0.6	1.3	3.9
Algonquin, Ill.	3.3	4.6	5.9	1.1	1.5	1.7	3.2
Plainfield, Ill.	1.6	1.0	5.8	1.4	0	1.1	1.7
South Beloit, Ill.	9.0	13.9	13.5	3.0	0.3	1.8	7.2
Rockford, Ill.	11.2	18.4	8.9	2.5	0.7	1.3	7.1

LITHOLOGIC AND PETROGRAPHIC INSPECTION

Insley's examination of the aggregates disclosed all were glacial mixed gravels, usually having been reworked by stream action. A number of different rock types were found in each sample; these included limestone, chert, quartz, quartzite, sandstone, individual feldspar particles, soft iron particles, graywacke, and igneous rock types. The igneous rock types were almost exclusively medium to coarse grained and included granite, diorite, and gabbro. No glassy volcanic or other types of effusive igneous rocks were observed, and no opal was detected. More than one half of the coarse fractions of each sample was composed of limestone. The lithologic analysis of the different sized fractions of gravel and sand from these was made following ASTM procedure as described in ASTM Recommended Practice C 295,[4] with the exceptions listed in the footnotes. The results of the analyses are given in Table I.

The weighted average shown in the last column of Table I was calculated from the respective weights of the sized fractions used in the lithologic analysis. Results are given specifically only for quartz, limestone, and chert with the group including the balance of material composed largely of plutonic, igneous rock, gneiss, sandstone, feldspar grains, and graywacke with an occasional claystone and soft iron particle in the larger sizes. Since no glassy volcanics or other potential reactive aggregate material was observed, the differentiation of this class was considered unnecessary

For all six of the samples investigated, there is generally a marked increase in the percentage of quartz particles with decreasing sieve size and, conversely, a decrease in both the percentage of chert and limestone particles.

---

[4] Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295 – 54), 1958 Book of ASTM Standards, Part 4, p. 573.

In addition to the chert count reported in Table I, a count was made of chert particles between sieve sizes $\frac{3}{8}$ in. to No. 4, and No. 8 to No. 16. The chert count for all sieve sizes examined is shown in Table II.

The chert count in the material retained on the $\frac{3}{8}$-in. sieve was greater than in any other sieve sizes and also was greater than the weighted average of all sieve sizes.

A count of the chert particles in heavy liquid fractions showed that all chert particles with the exception of a few containing considerable carbonate were in fractions lighter than specific gravity 2.65 and that 92 per cent of the earthy particles were in fractions ligher than specific gravity 2.60.

A lithologic count of chert particles, distinguishing earthy chert from waxy chert, in the sieve fraction No. 4 to $\frac{3}{8}$ in. was made on several fractions lighter than specific gravity 2.70 for the aggregate lots. It was observed that the more porous, earthy chert was higher in the lighter fractions.

Detailed petrographic examinations were made on 26 selected pebbles from the fraction coarser than $\frac{3}{8}$ in. and included two or more from each lot of gravel. The texture of the chert ranged from fine-grained granular quartz (like that typical of quartzite) through microgranular quartz to radiating microfibrous material of very fine, almost submicroscopic grain size.

Approximately 85 per cent of the silica material comprising the cherts had a refractive index higher than 1.540 and that silica material having a refractive index less than 1.540 had a typical radiating microfibrous texture.

It is evident from the lithologic and petrographic analyses that the only possible potentially reactive constituent of any of these gravels was the chert. Judging on the basis of commonly used criteria, three of the aggregates quite probably would be classed as potentially reactive inasmuch as the chert content is relatively high and, in part, consists of the microfibrous texture characteristic of chalcedony.

FIELD INSPECTION

The field trip embraced that area of Northern Illinois where it was most likely the suspect aggregates would be used in concrete construction. Some 25 existing structures, mostly bridges, dating from 1912 to the present time were inspected. Although there were little recorded data to establish what concrete materials were used in the structures, it is known that at least five of the structures used concrete containing aggregate of the type studied in the investigation and portland cement having an alkali content in excess of 0.6 per cent total as sodium oxide. Many of the concrete structures showed distress. However it appeared to be of the type resulting from chert or iron pop-outs caused by freezing and thawing, surface scaling of non-air-entraining concrete, and plastic and shrinkage cracking, attributable to concrete design, placing, and curing practices.

Although distress was very much in evidence in many of the structures, particularly those twenty years or more in age, the distress almost without exception could quite easily be recognized as being attributable to causes other than those associated with alkali expansion of concrete.

QUICK TEST FOR CHEMICAL REACTIVITY

Each of the fine and coarse aggregates from the six sources were tested according to ASTM Method C 289.[2] Using the criteria of this method for judging po-

TABLE III.—RESULTS OF CHEMICAL TEST FOR POTENTIAL REACTIVITY.

Aggregate Source (Illinois)	Silica Dissolved, millimoles per liter (S_c)	Reduction in Alkalinity, millimoles per liter (R_c)	ASTM Tentative Method C 289[a]	ASTM Specification C 33[a]
SANDS				
Forreston	145	210	innocuous	innocuous
Elgin	150	185	innocuous	innocuous
Algonquin	150	150	borderline	innocuous
Plainfield	165	155	borderline	deleterious
South Beloit	95	250	innocuous	innocuous
Rockford	110	215	innocuous	innocuous
GRAVELS				
Forreston	175	250	innocuous	innocuous
Elgin	165	180	innocuous	innocuous
Algonquin	165	145	deleterious	deleterious
Plainfield	185	150	deleterious	deleterious
South Beloit	165	250	innocuous	innocuous
Rockford	185	265	innocuous	innocuous

[a] If R_c is greater than 70, the aggregate is considered deleteriously reactive if S_c is greater than R_c.

TABLE IV.—RESULTS OF OSMOTIC CELL TESTS OF ILLINOIS AGGREGATES.

Test conducted on 50 to 100 mesh prepared sample in 3 per cent NaOH.

Aggregate Source (Illinois)	Total Flow, cm	
	Days	Flow
SANDS		
Forreston	31	0.4
Elgin	31	0.1
Algonquin	31	1.4
Plainfield	31	0.6
South Beloit	31	1.0
Rockford	22	−0.1
GRAVELS		
Forreston	31	1.1
Elgin	31	0.8
Algonquin	31	0.5
Plainfield	31	0.8
South Beloit	31	2.4
Rockford	22	0.6
OPAL		
	3¼	5.5

TABLE V.—MEASUREMENT OF LIMESTONE PRISM.

Source of Gravels	Expansion After 14 Days in 1*M* Sodium Hydroxide, per cent
Forreston, Ill.	−0.035
Elgin, Ill.	−0.042
Algonquin, Ill.	−0.037
Plainfield, Ill.	−0.036
South Beloit, Ill.	−0.059
Rockford, Ill.	−0.018
Iowa, Ont.	+1.230
Kingston, Ont.	+0.680

tential reactivity, the Algonquin and Plainfield fine aggregates were borderline; while both the Algonquin and Plainfield coarse aggregates would be deleterious (See Table III). Using ASTM Specification C 33[5] criteria, the Plainfield fine aggregate and the Algonquin and Plainfield coarse aggregates fell in the deleterious category (see Table III).

The Plainfield aggregate, by this criteria, would be the most deleterious; yet, the service record of the Plainfield aggregate shows no history of alkali expansion. These aggregates have also been used in the Long-Time Study at The Portland Cement Assn. without showing evidence

---

[5] Specifications for Concrete Aggregates (C 33 – 59), 1959 Supplement to Book of ASTM Standards, Part 4, p. 71.

TABLE VI.—EXPANSION OF MORTAR BARS AT 100 F AND 100 PER CENT RELATIVE HUMIDITY. ASTM METHOD C 227.

Aggregate Source (Illinois)	Cement	Length Change, per cent (+)						
	Alkali, per cent	1 month	2 months	3 months	6 months	9 months	1 yr	1½ yr
SANDS								
Forreston	0.17	−0.002	−0.002	−0.003	−0.001	−0.002	−0.001	−0.001
	0.57	0.006	0.013	0.013	0.017	0.018	0.018	0.020
	1.05	0.014	0.020	0.022	0.029	0.033	0.035	0.037
Elgin	0.17	−0.005	−0.004	−0.004	0	0.002	0.002	0.002
	0.57	0.008	0.011	0.013	0.017	0.018	0.018	0.021
	1.05	0.015	0.021	0.024	0.032	0.035	0.037	0.039
Algonquin	0.17	−0.003	−0.003	−0.003	−0.003	−0.002	−0.004	0.001
	0.57	0.012	0.012	0.012	0.015	0.018	0.016	0.022
	1.05	0.019	0.024	0.026	0.033	0.037	0.037	0.048
Plainfield	0.17	0.001	−0.002	−0.005	−0.006	−0.005	−0.005	0.002
	0.57	0.010	0.013	0.012	0.015	0.017	0.016	0.023
	1.05	0.018	0.019	0.019	0.025	0.028	0.032	0.040
South Beloit	0.17	−0.001	−0.003	−0.003	−0.001	−0.001	−0.005	−0.001
	0.57	0.011	0.012	0.013	0.013	0.013	0.015	0.020
	1.05	0.016	0.020	0.023	0.025	0.025	0.030	0.037
Rockford	0.17	0.003	0.005	0.004	0.008	0.009	0.008	0.010
	0.57	0.012	0.015	0.016	0.022	0.024	0.024	0.026
	1.05	0.019	0.023	0.025	0.034	0.039	0.039	0.042
GRAVELS								
Forreston	0.17	0.002	0.003	0.003	0.005	0.008	0.008	0.011
	0.57	0.012	0.015	0.016	0.020	0.024	0.024	0.028
	1.05	0.018	0.022	0.024	0.031	0.035	0.035	0.041
Elgin	0.17	−0.002	0	0	0.002	0.006	0.006	0.009
	0.57	0.010	0.015	0.016	0.021	0.026	0.026	0.030
	1.05	0.018	0.025	0.028	0.037	0.043	0.043	0.050
Algonquin	0.17	−0.005	−0.003	−0.003	0.001	0.004	0.003	0.008
	0.57	0.011	0.015	0.014	0.021	0.025	0.025	0.031
	1.05	0.016	0.023	0.026	0.034	0.040	0.040	0.046
Plainfield	0.17	−0.003	−0.001	−0.002	0	0.002	0.002	0.003
	0.57	0.010	0.016	0.016	0.020	0.023	0.024	0.026
	1.05	0.019	0.023	0.027	0.035	0.038	0.039	0.042
South Beloit	0.17	−0.004	−0.004	−0.003	0	0	0.001	0.002
	0.57	0.008	0.012	0.014	0.019	0.020	0.021	0.023
	1.05	0.015	0.019	0.022	0.029	0.031	0.033	0.036
Rockford	0.17	0	0.003	0.001	0.006	0.009	0.009	0.011
	0.57	0.010	0.017	0.013	0.020	0.021	0.022	0.025
	1.05	0.018	0.022	0.023	0.031	0.035	0.036	0.039

of alkali expansion distress in the 19 years the concrete has been under test.

OSMOTIC CELL TEST TECHNIQUE

To determine the comparative degree of alkali aggregate reaction, each of the fine and coarse aggregates were tested, using techniques described by Verbeck and Gramlick[6] (see Table IV).

The experimental procedure depends on the physical type of any deleterious reaction product, occurring primarily of the gel-like or solution-type. The amount of this deleterious product would be primarily reflected by the development of an osmotic pressure between a reference solution and a solution surrounding a reaction zone. The flows were measured in a simple osmotic cell, described in the above-mentioned paper. The flow rate of the reaction product, in this case, was compared with opal. By comparison, the total flow of the reaction products of the opal was from approximately 3 to 50 times that found with the aggregates, while the reaction time in days was 3¼ for the opal and from 22 to 31 for the aggregates.

MEASUREMENT OF LIMESTONE PRISMS AFTER EXPOSURE IN NaOH

To determine whether a parallel might exist between the type of reactivity found in some of the carbonate rocks of Iowa and Kingston, Ont., limestone fractions from the six gravels of Northern Illinois were cut into ¼ by ¼ by 1¼-in. prisms, saturated with water, immersed in 1*M* sodium hydroxide solution for two weeks, and measured for expansion. Each of the Northern Illinois gravels showed a decrease in volume after 14 days of exposure, while the Iowa and Kingston, Ont. aggregates showed expansions of 1.230 per cent and 0.680 per cent, respectively (see Table V). These results show that there is no similarity in alkali reaction between the carbonate gravels of Northern Illinois and those of Iowa or Kingston, Ont.

TABLE VII.—EAU CLAIRE, WIS., AGGREGATE PLUS ILLINOIS AGGREGATES.

Mortar bars at 100 F and 100 per cent relative humidity.

Cement, total alkali as Na_2O = 1.05 per cent.

Amounts, per cent		Expansion, per cent				
Eau Claire, Wis. Aggregate	Illinois Aggregate	1 month	2 months	3 months	6 months	9 months
ALGONQUIN—SAND						
0.....	100	0.019	0.024	0.026	0.033	0.037
25.....	75	0.012	0.019	0.023	0.026	0.030
50.....	50	0.011	0.018	0.025	0.029	0.035
75.....	25	0.011	0.018	0.025	0.030	0.036
100.....	0	0.009	0.017	0.028	0.037	0.042
ALGONQUIN—GRAVEL						
0.....	100	0.016	0.023	0.026	0.034	0.040
25.....	75	0.013	0.018	0.023	0.028	0.032
50.....	50	0.009	0.017	0.023	0.028	0.033
75.....	25	0.012	0.018	0.024	0.029	0.034
100.....	0	0.009	0.017	0.028	0.037	0.042
PLAINFIELD—SAND						
0.....	100	0.018	0.019	0.019	0.025	0.028
25.....	75	0.014	0.023	0.029	0.034	0.041
50.....	50	0.017	0.026	0.033	0.039	0.047
75.....	25	0.016	0.024	0.031	0.036	0.043
100.....	0	0.009	0.017	0.028	0.037	0.042
PLAINFIELD—GRAVEL						
0.....	100	0.019	0.023	0.027	0.035	0.038
25.....	75	0.016	0.024	0.031	0.036	0.042
50.....	50	0.017	0.024	0.032	0.037	0.041
75.....	25	0.018	0.024	0.030	0.034	0.037
100.....	0	0.009	0.017	0.028	0.037	0.042

MORTAR BAR EXPANSION[3]

Mortar bars for volume change testing were made, using each of the aggregates with cements having alkali contents of

---

[6] G. Verbeck and C. Gramlich, "Osmotic Studies and Hypothesis Concerning Alkali-Aggregate Reaction," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 1110 (1955).

TABLE VIII.—EXPANSION OF CONCRETE PRISMS IN INDICATED EXPOSURES.
Specimens: 3 by 3 by 11¼-in. prisms
Average Cement Content: 6¼ sacks per cu yd
Slump: 2½ to 3½ in.

Aggregate Source (Illinois)	Cement	Length Change, per cent (+)						
	Alkali, per cent	1 month	2 months	3 months	6 months	9 months	1 yr	1½ yr
100 F AND 100 PER CENT RELATIVE HUMIDITY								
Forreston	0.17	0.002	0.002	0.002	0.005	0.009	0.005	0.010
	0.57	0.007	0.008	0.010	0.013	0.016	0.012	0.019
	1.05	0.010	0.012	0.014	0.018	0.020	0.019	0.025
Elgin	0.17	0	0	0.002	0.005	0.006	0.004	0.009
	0.57	0.007	0.008	0.012	0.013	0.015	0.013	0.020
	1.05	0.010	0.011	0.015	0.019	0.022	0.021	0.028
Algonquin	0.17	0.001	0.001	0.003	0.005	0.006	0.006	0.008
	0.57	0.007	0.008	0.011	0.013	0.013	0.013	0.018
	1.05	0.009	0.011	0.015	0.018	0.019	0.020	0.025
Plainfield	0.17	0	0.001	0.004	0.004	0.005	0.004	0.008
	0.57	0.007	0.010	0.009	0.011	0.013	0.013	0.021
	1.05	0.012	0.012	0.013	0.015	0.020	0.019	0.027
South Beloit	0.17	0.001	0.002	0.006	0.006	0.006	0.005	0.010
	0.57	0.008	0.010	0.013	0.013	0.014	0.014	0.017
	1.05	0.010	0.012	0.016	0.019	0.020	0.019	0.025
Rockford	0.17	−0.007	−0.006	−0.006	0.001	0.001	0	0
	0.57	0.001	0.003	0.003	0.007	0.010	0.009	0.009
	1.05	0.004	0.006	0.006	0.014	0.017	0.016	0.016
73 F AND 100 PER CENT RELATIVE HUMIDITY								
Forreston	0.17	0.002	0.001	0.001	0.007	0.007	0.007	0.008
	0.57	0.002	0.001	0.002	0.008	0.008	0.006	0.011
	1.05	0.005	0.005	0.006	0.014	0.014	0.013	0.019
Elgin	0.17	0.001	0.001	0.002	0.007	0.007	0.004	0.009
	0.57	0.005	0.004	0.005	0.012	0.011	0.010	0.014
	1.05	0.005	0.006	0.008	0.016	0.016	0.015	0.020
Algonquin	0.17	0.005	0.003	0.005	0.008	0.008	0.007	0.011
	0.57	0.003	0.003	0.006	0.009	0.009	0.008	0.013
	1.05	0.005	0.005	0.007	0.014	0.012	0.011	0.018
Plainfield	0.17	0.002	0.001	0.002	0.006	0.004	0.004	0.009
	0.57	0.004	0.003	0.004	0.010	0.009	0.008	0.014
	1.05	0.005	0.004	0.005	0.012	0.012	0.011	0.017
South Beloit	0.17	0.003	0.002	0.003	0.009	0.006	0.005	0.009
	0.57	0.004	0.003	0.005	0.010	0.010	0.008	0.014
	1.05	0.006	0.006	0.009	0.012	0.014	0.014	0.019
Rockford	0.17	0.002	0.004	0.005	0.005	0.008	0.005	0.005
	0.57	0.003	0.006	0.007	0.008	0.010	0.009	0.009
	1.05	0.005	0.009	0.011	0.013	0.016	0.015	0.015

0.17, 0.57, and 1.05 per cent, respectively. Test procedures and storage conditions, prescribed by ASTM Method C 227, were followed. The length changes were recorded at 1, 2, 3, 6, 9 months, 1 yr, and 1½ yr. The maximum expansion was 0.05 per cent for the Elgin, Ill. gravel, and the average expansion for all coarse and fine aggregate mortar bars, using cement with an alkali content of 1.05 per cent was 0.041 per cent at the end of 1½ yr (see Table VI).

This test when made with a cement in excess of 0.8 per cent, expressed as sodium oxide, will furnish information on the likelihood of harmful reaction occurring. Combinations of aggregate and cement which have produced excessive expansions in this test usually should be considered potentially reactive. While the line of demarcation between nonreactive and reactive combinations is not clearly defined, it exceeds 0.05 per cent at 3 months or 0.10 per cent at 6 months.

In view of earlier published work which showed mortar bar expansion might be directly influenced by a percentage relationship between the reactive mineral and the alkali content, the mortar bar test was repeated, using the two fine and two coarse aggregates showing the greatest potential reactivity in Method C 289 with varying increments of an aggregate from Eau Claire, Wis., which was known to be nonalkali reactive. Sixteen mortar bars were made with cement having an alkali content of 1.05 per cent total as Na_2O, in combinations with the Illinois and Wisconsin aggregates. The aggregate combinations were made, decreasing the Illinois and increasing the Wisconsin aggregates, in increments of 25 per cent. The results which are shown on Table VII illustrate that the mortar bar expansion values of the Algonquin and Plainfield aggregates were neither increased nor decreased significantly with use of varying quantities of an aggregate known to be nonreactive.

CONCRETE PRISMS EXPANSION

To determine whether the expansion behavior of concrete would differ from that of the mortar bars, 3 by 3 by 11¼ in. concrete prisms were cast, using each fine and coarse aggregates tested in the mortar bar program. To simulate the commercial product, the material retained on 1½-in. sieve was crushed, screened, and recombined with the original No. 4 to 1½ in. The entire sample was then separated into three fractions. In proportioning the materials for the concrete prisms, the three fractions were recombined so as to maintain the percentage of each fraction to the same grading yielded in the processed product. A 6¼-sack cement factor was used and the slump maintained between 2½ in. and 3½ in. Concrete prisms were cast, using the same three cements used in the mortar bar test, and measurements were taken at the same time intervals, up to 1½ yr. Exposure temperatures were at 73 F and 100 F, and relative humidity was maintained at 100 per cent under both temperature conditions (see Table VIII).

At the end of 1½ yr, the maximum expansion for the specimens manufactured with 1.05 per cent alkali, stored at 100 F, was 0.028 per cent and the average expansion for all bars with this cement was 0.024 per cent.

With the same cement, at the end of 1½ yr, the specimens exposed at 73 F showed a maximum expansion of 0.020 per cent, with an average expansion of 0.018 per cent.

The difference in expansion between the prisms in which the 0.17 per cent alkali cement and the 1.05 per cent alkali cement had been used averaged 0.016 per cent for the prisms stored at 100 F, and 0.009 per cent for the prisms stored at 73 F.

VOLUME CHANGE IN CONCRETE STRUCTURES

Permission was obtained from The Illinois State Toll Highway Commission to install gage points for length change measurement on certain structures of the Northern Illinois Toll Road, and in June and July of 1958, points were placed in eight structures of the type most likely to display alkali expansion, at an early age, if deleterious reaction occurred. The point span was approximately 100 in. and measurements were made periodically through April 27, 1960. The date of concrete placement, in the structures under test, ranged from February to November, 1957. Of 16 positions in eight structures, there has been no abnormal volume change in any position. Fourteen of the positions show volume changes between −0.009 in. and +0.005 in.; the two remaining positions measured increases of +0.008 in. and +0.013 in. The positions showing the highest volume change values were at a wingwall and bridge abutment and presented the most advantageous area for expansion in that there was a constant source of water keeping the concrete in a continuous moist condition (see Table IX).

TABLE IX.—EXPANSION MEASUREMENTS ON ILLINOIS TOLL HIGHWAY.

Structure	Structure Number	Aggregate Source	Cement Alkali Total as Na$_2$O, per cent	Average Change, in.	Measurement Period, months
Bridge	N-1-14	Rockford	0.96	−0.002	22
Bridge crashwall	N-1-11	South Beloit	0.94	−0.002	22
Wingwall	N-1-3	South Beloit	0.94	+0.013	16[a]
Abutment	N-1-2	South Beloit	0.94	+0.006	22
Box culvert	N-2-10	South Beloit	0.94	−0.005	22
Bridge pier stems	N-3-1	Rockford	0.94	−0.000	12[a]
Bridge abutment	N-3-7	Rockford	0.87 to 1.04	+0.008	11[a]
Railroad bridge	N-1-9	South Beloit	0.50 to 0.55	−0.009	22

[a] Points broken or unable to read.

The aggregates used in the structures were from the Beloit and Rockford deposits, and the alkali content of the cement used in seven of the structures ranged from 0.87 per cent to 1.04 per cent. The cement used in the remaining structure under study contained alkali of the order of 0.50 to 0.55 per cent total as Na$_2$O, and the aggregate used was from the Beloit deposit.

Considering that the maximum volume change after up to 22 months of study was of the order of 0.01 per cent, it would appear that expansion due to alkali reactivity is not likely to be a source of distress. Additionally, these data would tend to support the historical background of the service performance of the aggregates from this geological area.

Because surface cracking in the deck of the Green Street Bridge in McHenry, Illinois was considered by the investigators to be due possibly to alkali expansion, gage points were also installed in six positions of the deck of this structure. The measurements in five of the positions, after a 2-yr interval, show no expansion. Data were not obtainable on the remaining position as it was damaged by a snow-plowing operation. In view of these findings, after more than 2 yr of observation and measurements, it would appear that alkali aggregate expansion could also be ruled out as a cause of the cracking on the deck of the Green St. Bridge.

SUMMARY

In summary, it would appear that we might draw two conclusions from the investigation. The first is that the degree of alkali reactivity between the cement alkalies and the suspect aggregates is not sufficient to cause alkali expansion in the concrete, nor is it responsible for the distress occurring in those structures suspected of showing expansion cracking. The second conclusion would appear to be that the use of the ASTM Chemical Test for Reactivity (Method C 289) and the present criteria for judging potential alkali reactivity, petrographically, are not a reliable means of determining whether or not a given aggregate is likely to be reactive to the degree that it will cause expansion or distress.

The Algonquin and Plainfield aggregates are suspect, using information obtained from the ASTM Chemical Test for Reactivity, as criteria for judging reactivity. On the other hand, Insley's work suggests that the Algonquin and Plainfield aggregates probably are not reactive. However, the Forreston, South Beloit, and Rockford aggregates would definitely be suspect, inasmuch as chalcedony was detected, and they contained chert in excess of 5 per cent. Although the other tests on the aggregates cannot very well be used as criteria for judging alkali reactivity, the tests do show that when compared to opal, and the Iowa, and Kingston, Ontario carbonate stones, the degree of reactivity is not significant.

The matter is further confused by the fact that the volume changes in the mortar bars, concrete prisms, and structures on the Northern Illinois Toll Road System failed to agree with either the ASTM Chemical Test for Reactivity or the petrographic analysis.

As the chemical test and petrographic analysis are, at present, the most rapid means of obtaining evidence of reactivity, there probably is a natural tendency for specification writers to rely on these methods for determining whether an aggregate is deleteriously reactive. This investigation points out, we believe, that a perfectly satisfactory aggregate might erroneously be judged alkali reactive if the judgment is made on the basis of these criteria. In fact, had it not been for the record of service performance of these aggregates, the early work in the investigation would have led us to believe the aggregates were deleteriously alkali reactive.

PERFORMANCE TESTS OF FIELD CONCRETE MIXERS*

BY I. NARROW[1]

SYNOPSIS

A study was made to determine the performance of two 4-cu yd stationary concrete mixers used for airfield pavement construction at Clinton County Air Force Base, Ohio. Evaluation of mixer performance was based on tests for uniformity of composition, workability and strength of concrete samples from the front, middle and rear of the mixers. Five different mixing periods were used, ranging from 135 sec to 45 sec after the materials had entered the mixer drum.

The tests showed appreciable difference in performance of the two mixers. One mixer produced uniformly mixed concrete only when the maximum mixing time of 135 sec. was used, while the other mixer showed satisfactory performance with a mixing time as low as 75 sec. Conclusions are made regarding the suitability of the different tests for evaluating mixer performance.

Corps of Engineers specifications require that minimum mixing time for stationary and paving mixers shall be not less than 1 min for mixers having a capacity of 1 cu yd, with an increase in mixing time of 15 sec for each additional $\frac{1}{2}$ cu yd or fraction thereof of concrete mixed when larger mixers are used.

The question of mixing time was raised by contractors proposing to use central-plant mixing with large-size stationary mixers, for airfield pavement construction at Clinton County Air Force Base. They contended that the mixing time requirement was excessive for large mixers and would unnecessarily increase the cost of central mixing so that it would not be competitive with paving mixers. As a result, the specifications for this project were modified to require 15 sec increase in mixing time for each additional cubic yard or fraction thereof of mixed concrete when the batch exceeded 1 cu yd in volume. This conforms with the mixing time requirements for stationary mixers specified in the ASTM Standard Specification for Ready-Mixed Concrete (C 94).[2] This change appreciably reduced the mixing time for large-size stationary mixers but did not affect mixing time for paving mixers.

The purpose of this study was to evaluate the performance of the concrete mixers used for the airfield pavement construction at this project. Since no standard test procedures nor mixer performance requirements were available,

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Chief, Concrete Division, U. S. Army Engineer Division Laboratories, Ohio River, Cincinnati, Ohio.

[2] Standard Specification for Ready-Mixed Concrete (C 94 – 58), 1958 Book of ASTM Standards, Part 4, p. 600.

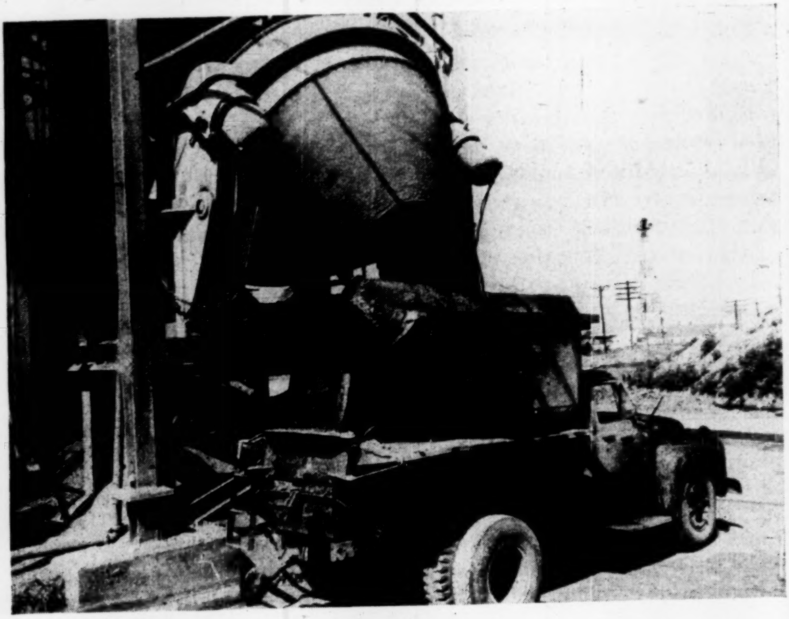


FIG. 1.—Mixer Discharging Concrete.

FIG. 2.—General View of Test Area.

the test program was planned to include a large number of tests that would provide data suitable for evaluating the performance of the mixers. The evaluation of the test data and test procedures also was intended to provide information useful for establishing standard mixer performance requirements and test procedures.

The study was limited to the specific mixers, concrete mix, and mixing conditions that were being used for the airfield pavement construction. The evaluation of mixer performance was based on uniformity of the concrete mix throughout the batch. Samples were taken from the first, middle, and last portions of the batch as discharged from the mixer. Tests were made to determine uniformity of composition, cement content, water content, air content, workability, and strength of the concrete samples from the batch.

This paper presents the results of the tests conducted and reviews the test procedures used. The performance of the mixers for the mixing periods used is indicated on the basis of tentative limits selected for evaluation of the test results. The suitability of the tests made for evaluation of mixer performance is discussed.

DESCRIPTION OF CONCRETE PLANT

Equipment:

The batching plant consisted of a three-compartment aggregate bin and a cement bin, with automatic batching equipment and individual weighing hoppers. A dry-batch hopper was located beneath the weighing hoppers to collect the weighed materials prior to charging the mixers. Water was weighed separately, and a measured quantity of air-entraining agent was charged into the mixer with the water.

The plant was equipped with two identical 4-cu yd tilting mixers which were used to mix 4.4 cu yd of concrete per batch. The mixers were set facing two opposite sides of the plant and were charged from the rear through fixed charging chutes leading from the dry-batch hopper. The direction of the charging of the dry materials was controlled by means of a "flap" gate in the dry-batch hopper. The tilting mixers were discharged directly into Dumpcrete trucks used for hauling the concrete to the paving site. Figure 1 shows a view of the mixer discharging concrete into a Dumpcrete hauling truck.

Batching and Mixing:

The batching of the concrete materials was automatic after the action was initiated by the operator. However, the weighing hoppers did not have the capacity to handle the quantities of materials required for the 4.4 cu yd of concrete being mixed in each batch. It was therefore necessary to weigh the batches of materials in two portions. The scales were set to weigh one half of the required batch weights of each aggregate and cement. The first half of the batch was weighed and deposited in the dry-batch hopper. The remainder of the batch was then weighed and held in the weighing hoppers. The entire quantity of water required for the concrete batch was weighed in a separate water batcher. When the mixer charging cycle was started, the material entered the mixer from the dry-batch hopper. However, a separate button had to be pushed to discharge the remainder of the batched materials from the weighing hoppers. This action required only a few seconds and the flow of material from the dry-batch hopper and weighing hoppers into the mixer generally was continuous. Water was charged directly into the mixer from the water batcher.

The mixing time was set on a time clock, and the timing of the mixing was started when the mixer charging cycle was started. For the test batches, the mixers were discharged immediately after the time mixing time set on the time clock had elapsed. The discharge of the mixer was controlled by the operator as necessary for the sampling of the concrete.

TEST PROGRAM

Concrete Mix:

The concrete used for the pavement construction had the following composition:

Coarse aggregate—crushed limestone, 2-in. to 1-in., and 1-in. to No. 4 sizes;
Fine aggregate—natural sand;
Cement content—6.0 bags per cu yd;
Water - cement ratio—4¾ gal per bag;
Air content—about 4 per cent; and
Slump—1 to 2 in.

Tests:

The tests were conducted on the two concrete mixers. Five sets of concrete samples were taken for each of five mixing periods, as follows: 135 sec, 105 sec, 90 sec, 75 sec, and 45 sec. Each set consisted of three samples; one each from the first tenth, middle, and last tenth of the batch. Each sample contained about 2 cu ft of concrete. The following tests were conducted:

Slump of concrete;
Air content of concrete and mortar;
Unit weight of concrete and mortar;
Percentage of coarse aggregate in concrete;
Cement content of mortar;
Water content of mortar; and
Compressive strength of concrete.

All tests, except for cement content, water content, and compressive strength, were made on each sample taken. The cement and water-content tests were made on three sets of samples, and the compressive-strength tests were made on the other two of the five sets of samples taken for each mixer test condition.

Test Procedures:

Sampling.—Test samples were obtained after the mixed concrete had been discharged into the Dumpcrete hauling trucks. To facilitate the sampling, the paving contractor had constructed a platform of suitable height so that the concrete samples could be handed from the trucks onto the platform (Fig. 2). After the concrete had been mixed for the required time, the first one tenth of the batch (about 10 cu ft) was discharged into an empty truck. Additional concrete from the batch was then discharged into another empty truck until only about one tenth of the batch of concrete remained in the mixer. The last one tenth of the batch was then discharged into a third empty hauling truck. The concrete mixer was rotated when the concrete was being discharged into the trucks but was stopped when concrete was not being discharged. Each truck, in turn, was moved to the sampling platform where the concrete samples were taken.

A representative sample of the concrete in the trucks was obtained by shoveling from several different locations. The concrete was placed into metal pails and transferred to a large metal pan. The sample was covered with damp burlap until the tests were made. Before the individual test samples were taken, the large sample was remixed with a shovel just sufficiently to insure uniform distribution of the materials.

Slump.—The slump test was made on each concrete sample in accordance with standard procedures.

Air Content.—The air content of the concrete was determined both with a pressure meter and gravimetrically from the unit weight measurements. The concrete was consolidated in the measuring bowl by hand-rodding and by tapping the bowl.

The air content of the mortar portion

of the concrete was computed as follows:

$$A_m = \frac{A_s \times V_s}{V_s - V_g}$$

where:

A_m = air content of mortar, per cent,
A_s = air content of concrete sample, per cent,
V_s = volume of concrete sample, cu ft,
V_g = volume of coarse aggregate, cu ft

$$\left(V_g = \frac{W_g}{G \times W_w}\right),$$

W_g = weight of coarse aggregate in sample, lb,
G = specific gravity of coarse aggregate, and
W_w = unit weight of water, lb per cu ft.

Unit Weight.—The unit weight of the concrete was determined on the sample that was used for measuring air content. Prior to conducting the tests, the measuring bowl of the pressure meter was accurately calibrated for volume. The weight of the concrete was determined after consolidation in the measuring bowl for the air content test, and the unit weight of the concrete was computed by dividing the concrete weight by the volume of the measuring bowl.

Air-free unit weight of the mortar was computed from the data obtained in the tests for unit weight and air content of the concrete and the percentage of coarse aggregate in the concrete. The following formula was used for these computations:

$$W_m = \frac{W_s - W_g}{V_s - V_a - V_g}$$

where:

W_m = unit weight of mortar (air-free), lb per cu ft,
W_s = weight of concrete sample, lb,
W_g = weight of coarse aggregate in sample, lb,
V_s = volume of concrete sample, cu ft,
V_a = volume of air in sample, cu ft = $V_s \times$ per cent air,
V_g = volume of coarse aggregate, cu ft

$$\left(V_g = \frac{W_g}{G \times W_w}\right),$$

G = specific gravity of coarse aggregate, and
W_w = unit weight of water, lb per cu ft.

Percentage of Coarse Aggregate.—The weight and percentage of coarse aggregate in the concrete were determined by washing the concrete sample used for the air-content test over a No. 4 sieve. The coarse aggregate was surface dried by wiping with cloths, and then weighed. The percentage of coarse aggregate was based on the weight of the concrete sample.

Cement Content.—The cement content of the mortar was determined essentially in accordance with Corps of Engineers Method CRD-C72,[3] except that the sample was separated from the concrete mix by sieving over a No. 4 sieve. This test method is based on the method proposed by Hime and Willis.[4]

A representative portion of the concrete sample was sieved over a No. 4 sieve to provide about 15 lb of mortar. Two 250-g samples obtained from this mortar batch were dried and used for the cement content determinations. The two dried samples were combined and pulverized with a mortar and pestle until all material passed a No. 30 sieve. Two 20-g samples were obtained from the pulverized mortar and placed into separate 40-ml centrifuge tubes. Separation of the cement from the mortar in the centrifuge was made in accordance with the standard procedure.

---

[3] Method of Test for Cement Content of Freshly Mixed Concrete (CRD - C72), Handbook for Concrete and Cement, Corps of Engineers, June 1959.

[4] W. G. Hime and R. A. Willis, "A Method for the Determination of the Cement Content of Plastic Concrete," ASTM BULLETIN, No. 209, Oct., 1955, p. 37 (TP 191).

The weight of material at the bottom of the tube below the heavy liquid was determined after the final period of centrifuging. The procedure followed was to carefully pour off the top layer of material (sand portion) and most of the heavy liquid. Due to the compaction during centrifuging, the cement in the lower portion of the tube did not flow with the liquid, and no difficulty was encountered in retaining this material in the tube. Acetone was added to the tube, and the material was thoroughly agitated to remove the heavy liquid. The material in the tube was then dried and weighed.

Examination of the dried material from the bottom of the tube disclosed that some heavy sand minerals were included with the cement. It was therefore necessary to make a correction for the heavy sand material included with the cement. This correction factor was determined by centrifuging samples of the sand using the same heavy liquid as for the cement-content tests. These tests indicated that 2.2 per cent of the sand was composed of materials that would sink in the separation liquid, and this correction was used for all cement content determinations. The cement content was computed as a percentage of the initial weight of the dried mortar sample used in the test.

Water Content of Mortar.—The water content of the mortar was determined by measuring the weight loss during drying of the 250-g mortar samples used for determining cement content.

Compressive Strength.—Three 6 by 12 in. cylinders were cast from each sample tested for compressive strength. Preparation, moist-curing and testing of the specimens all were in accordance with standard procedures. All strength tests were made when the concrete specimens were 28 days old.

TEST RESULTS AND DISCUSSION

Sampling:

Because of the arrangement for charging the concrete mixers, which prevented access to the rear end of the drum, it was impracticable to obtain the test samples directly from the mixers. The sampling, therefore, was done after the individual portions of the batch (first tenth, middle portion, and last tenth) were discharged into separate hauling trucks. The platform arrangement for handling the test samples from the hauling trucks was very satisfactory.

For the tilting mixers used it was difficult to control the discharge of the concrete so that the portions from the front and rear of the mixer contained approximately the required 10 per cent of the batch. This was especially true for the last portion of the batch as there was no way to determine how much concrete remained in the mixer during discharge. In several instances, the batch intended for testing was not used because the first or last portion of the batch did not contain the proper amount of concrete.

There was no noticeable segregation in the different portions of the batch for any of the mixing times used. This is attributed to the comparatively rich mix used and to the full flow of the concrete during discharge from the tilting mixers. With this type of discharge, there is some question whether the three portions of the batch as discharged into the trucks actually represent concrete from the front, center, and rear of the mixer. However, there was no way to determine the flow pattern of the concrete from the mixers.

Mixing Time:

The mixing time set on the time clock was the gross time including the time required for charging the mixer. Several time checks made during the study indi-

TABLE I.—SUMMARY AND ANALYSIS OF TEST DATA.

Mixing Time, sec	Location in Mixer	Concrete										Mortar													
		Slump[a]		Air Content[a]		Coarse Aggregate[a]		Unit Weight[a]		28 Day Strength[b]		Cement Content,[b]		Water Content[c]		Air Content[a]		Unit Weight[a] (Air-Free)		Water - Cement[a] Ratio		Sand - Cement Ratio		Strength - Cement - Content[b] Ratio	
		Test, in.	Variation,[d] in.	Test, per cent	Variation,[e] per cent	Test, per cent	Variation,[e] per cent	Test, lb/per cu ft	Variation,[e] per cent	Test, psi	Variation,[e] per cent	Test, per cent	Variation,[e] per cent	Test, per cent	Variation,[e] per cent	Computed, per cent	Variation,[e] per cent	Computed, per cent	Variation,[e] per cent	Computed by Weight	Variation, per cent	Computed by Weight	Variation,[e] per cent	Computed Factor	Variation,[e] per cent
MIXER No. 1																									
135....	front	2½		4.8		55.7		145.4		5565		29.6		13.9		9.4		140.8		0.470		2.38		188	
	middle	3	½	4.6	8.7	53.2	4.9	144.8	0.7	5455	2.8	29.3	1.4	13.7	1.5	8.8	6.6	140.8	0.2	0.468	0.4	2.41	1.7	186	3.2
	rear	3		4.4		55.9		145.8		5410		29.7		13.9		8.8		141.1		0.468		2.37		182	
105....	front	2¾		4.2		55.6		145.8		6415		30.7		13.6		8.4		140.8		0.443		2.26		209	
	middle	3¼	¾	4.3	4.6	56.5	2.3	145.2	0.4	4840	28.3	24.1	24.3	13.3	4.4	8.9	6.8	139.4	1.0	0.551	21.3	3.15	32.6	201	4.4
	rear	3½		4.4		56.9		145.4		5285		26.5		13.9		9.0		140.0		0.525		2.77		200	
75....	front	1¾		3.7		53.3		147.0		7580		34.3		13.1		7.1		142.4		0.382		1.92		221	
	middle	1¾	½	4.2	12.8	55.0	4.6	146.3	0.6	5645	30.4	24.7	33.9	12.4	5.5	8.3	15.8	141.8	0.4	0.502	26.5	3.05	44.8	228	3.1
	rear	1¼		3.7		55.8		147.2		5905		25.9		12.4		7.3		141.9		0.479		2.86		228	
45....	front	2		3.6		53.3		146.8		7120		35.4		14.3		6.9		142.2		0.404		1.83		201	
	middle	1¾	½	4.6	24.4	54.4	2.0	145.9	0.6	5380	28.8	23.9	41.3	13.0	12.8	9.0	26.6	142.5	0.7	0.544	28.5	3.18	49.5	225	15.5
	rear	1½		4.0		54.4		146.2		5650		24.0		12.6		7.9		141.5		0.525		3.17		235	
MIXER No. 2																									
135....	front	2¼		4.8		55.9		145.2		5105		30.4		14.0		9.6		141.5		0.460		2.29		168	
	middle	1¾	¾	4.6	8.7	54.2	3.1	145.9	0.8	5375	9.6	29.2	8.2	13.5	7.4	8.9	8.8	142.9	1.0	0.462	0.9	2.42	11.5	184	17.4
	rear	1½		4.4		55.6		146.3		5620		28.0		13.0		8.8		142.4		0.464		2.57		200	
105....	front	1½		3.6		54.5		148.2		6575		29.3		13.2		7.2		144.1		0.450		2.41		224	
	middle	1¼	¾	3.5	5.7	54.5	1.5	148.8	0.9	6345	5.1	32.6	13.2	12.9	3.0	7.0	4.3	145.1	1.1	0.396	13.0	2.07	19.4	195	14.1
	rear	¾		3.4		55.3		149.5		6680		33.5		13.3		6.9		145.7		0.397		1.99		200	
90....	front	2½		5.4		53.1		143.8		4785		31.2		13.4		10.2		141.7		0.430		2.20		154	
	middle	2¼	1¼	5.0	16.0	54.1	3.5	145.3	1.7	4930	3.0	32.5	4.1	13.6	2.2	9.7	11.3	142.7	1.1	0.418	4.0	2.08	5.6	152	1.3
	rear	1¼		4.6		55.0		146.2		4875		32.2		13.3		9.1		143.2		0.413		2.11		152	
75....	front	1½		3.5		56.5		148.4		6210		30.7		13.6		7.2		143.5		0.443		2.26		202	
	middle	1¼	¼	3.9	10.5	54.5	4.9	147.8	0.4	5855	6.4	29.7	5.6	13.5	0.7	7.7	6.7	144.8	0.9	0.454	5.4	2.37	7.9	197	8.2
	rear	1½		3.9		53.8		147.9		5825		31.4		13.5		7.7		144.7		0.430		2.19		186	
45....	front	1½		3.8		58.9		147.5		5155		36.5		14.5		8.2		141.8		0.397		1.74		141	
	middle	1¼	¼	4.1	7.5	54.5	9.0	147.7	0.4	5540	7.1	26.6	32.5	12.8	12.6	8.1	5.0	144.6	2.0	0.482	19.0	2.76	43.6	208	37.0
	rear	1½		4.0		53.9		147.1		5500		28.5		13.2		7.8		143.5		0.463		2.51		193	
Tentative limits...			1		20.0		8.0		1.5		10.0		10.0		10.0		20.0		1.5		10.0		10.0		

[a] Each value is average of 5 tests.
[b] Each value is average of 6 tests.
[c] Each value is average of 3 tests.
[d] Maximum value − minimum value.
[e] $\frac{\text{Maximum value} - \text{minimum value}}{\text{Average of 3 values}} \times 100.$

cated that it took about 20 seconds for all of the solid materials to enter the mixer drum. Some additional mixing occurred during discharge of the batch from the mixer, which required about 10 sec. The mixing time shown for each test batch was the net time after charging of the mixer had been completed until the mixer was released for discharge. The maximum mixing time (135 sec) was that used at the project when the tests were started. The reductions in mixing time were made for 30-sec intervals, except that an additional mixing time of 90 sec was used for one mixer.

Although every effort was made to maintain uniform batching, mixing, and sampling conditions, it is probable that small variations in mixing time did occur. Any delay during charging of the mixer would reduce the net mixing time, while delay after the mixing cycle was completed would increase the time of mixing. Also, for the sampling procedure used, the first portion of the concrete batch had the least mixing, and the middle and last portions were mixed progressively slightly longer. It is believed that the mixing time generally was within about 5 sec of the scheduled time. Closer control of the mixing time cannot be maintained for the mixing and sampling procedures used in these tests. It is believed that the small variations that may have occurred in individual mixing times had no appreciable effect for the large range in mixing time covered by the test program.

Test Results:

A summary of the test results for each mixer and mixing time is given in Table I. This table shows average values for each test and variations in average test results for the samples from the front, center, and rear of the mixer. Also shown are computations made for evaluating uniformity of the test results, as well as tentative limits for variation in test results for use in determining mixer performance. The following discussions of the test results are based on data shown in Table I.

Slump.—The average slumps of the concrete samples generally ranged between 1 and 3 in. Except for a few batches, the individual slump values were within the same range. The variations in slump generally were between $\frac{1}{4}$ and $\frac{1}{2}$ in. These differences were approximately the same for all mixing periods. The variations in slump are within the range that may be expected for uniformly well-mixed concrete.

The slump tests indicate that there was no difference in mixing efficiency of the concrete mixers for any of the mixing periods used. However, it is believed that the slump test was not sufficiently sensitive for evaluating mixer performance for the low-slump concrete used.

Air Content.—Air content of the concrete samples determined with a pressure meter agreed very closely with the values obtained gravimetrically from the unit-weight measurements. Table I shows a summary of the pressure meter test results. The average air content of the concrete was about 4 per cent, and the maximum range in average air content was between 3.5 and 5.4 per cent. Mixer No. 1 with a 45-sec mixing time had a maximum variation in air content of 1.0 per cent. In all other instances, the variations in air content were 0.5 per cent or less. In general, the variations in air content were at a minimum for the longer mixing times (135 and 105 sec), and tended to increase slightly for the shorter mixing times. However, all of the variations were comparatively small, and the tests indicate satisfactory uniformity of air entrainment for all mixing periods. It is believed that a practical limit for maximum variation in air content would be 1.0 per cent of the measured air content, or about 20 per cent variation from the average value.

Air contents of the mortar fractions of the concrete samples were determined in order that the evaluation of the test results would not be influenced by possible variation in coarse-aggregate content due to sampling. The mortar air contents shown in Table I were computed from the pressure meter air contents. These data show essentially the same order of variation and trend in variation for the different mixing times as those indicated by the tests on the concrete samples.

For the concrete mix and maximum aggregate size used in these tests, the variation in coarse aggregate, due to sampling, were comparatively small, and there was little preference between the air test on concrete or mortar. However, for lean concrete mixes, or when large-size coarse aggregate is used, it is probable that the mortar air test would be more reliable than the concrete test for determining variation in air content. In these tests, the mortar-air content was computed from other available test data for the concrete. It would be entirely feasible, and in many instances more practicable, to determine air content directly on the mortar samples.

Unit Weight.—The maximum variation in unit weight of the concrete within the batch generally was less than 1 per cent. Variation in weight between the different portions of the batch generally were less than 1 lb per cu ft. The variations in unit weight between the different batches are somewhat greater. However, a comparison of these unit weights with the air contents of the concrete shows that the variations in unit weight are directly influenced by the quantity of air entrained in the concrete. Variations in unit weight within the batch also would be influenced by variation in air content but to a lesser degree because of the smaller variation in air content. The test results indicate that there was no difference in uniformity of unit weight of the concrete within the batches for any of the mixing periods used in this study.

The unit weight of the mortar, on an air-free basis, was computed from the data on unit weight, air content, and percentage of coarse aggregate in the concrete sample. For these values, the effects of variations in air content and percentage of coarse aggregate are eliminated, and the results should provide satisfactory indication of the composition of the mortar within the batch. The maximum variation in unit weight within the batches ranged from 0.2 to 1.1 per cent, except for mixer No. 2 with a 45-sec mixing time, which had a maximum variation of 2.0 per cent. The U. S. Bureau of Reclamation Concrete Manual[5] specifies a maximum variation in samples from front and rear of the mixer of not more than 0.8 per cent from the average of the two mortar weights. However, it is considered preferable to evaluate mixer performance on the basis of maximum variation between the samples from the front, center, and rear of the mixer. In this case the limit should be about twice that specified by the Bureau of Reclamation, and a maximum limit of 1.5 per cent has been used for this study. Only mixer No. 2 with a 45-sec mixing time failed to meet this limit.

Coarse Aggregate Content.—Percentages of coarse aggregate are shown in terms of the weight of the concrete samples. The Bureau of Reclamation Concrete Manual[5] specifies that the weight of coarse aggregate per cubic foot of concrete in the front and rear of the mixer shall not vary more than 5 per cent from the average of the two weights. The variation in terms of percentage of coarse aggregate would be essentially the same as on a weight basis. For evaluation of mixer performance based on maximum variation between front, center, and rear of the mixer, the limit has been increased to 8.0 per cent. Only mixer No. 2 with a 45-sec mixing time failed to meet this limit. In all other instances the maximum variations were within the 8-per cent limit.

---

[5] Concrete Manual, U. S. Bureau of Reclamation, Denver, Colo., Sixth Edition (1955).

Water Content of Mortar.—Variations in water content within the batch generally were several times greater for the 45-sec mixing time than for the longer mixing periods. For mixer No. 1 the minimum variation in moisture content was obtained with the longest mixing time, and the variation increased as the mixing time was reduced. However, for mixer No. 2 the minimum variation occurred for the 75-sec mixing time and the variations were progressively greater for the longer mixing times. An 8-percent limit for variation in water content has been used for evaluation of the test results. On this basis, only the 45-sec mixing time for both mixers did not produce satisfactory distribution of the mixing water throughout the batch.

Compressive Strength.—The compressive strength data indicate variable performance for the two mixers used in the test. Although no limits for variation in compressive strength have been established, the general experience with this test indicates that a variation in strength of the concrete from different portions of the batch of 10 per cent or less may be considered satisfactory. On this basis, the test results indicate that mixer No. 2 produced uniform mixing of the concrete batches for all mixing periods used. The variation in strength between batches may be associated with variations in air content, water content, and slump and are not pertinent to the evaluation of the mixers.

For mixer No. 1, uniform strengths were obtained only for the maximum mixing time used, and large variations in strength were obtained for all shorter mixing periods. In each instance, the concrete from the front end of the mixer had abnormally high strength, while the concrete from the middle and rear of the mixer had uniform but appreciably lower strength. The strength of these concrete batches, except for the front portion of the batches, was within the strength range for the concrete mix used.

The test results indicate that performance of mixer No. 2 was satisfactory for all mixing periods used. Mixer No. 1 provided satisfactory mixing of the concrete only at the maximum mixing period of 135 sec.

Cement Content.—Results of the cement-content tests are shown as percentages of the dry weight of the mortar samples used in the tests. The test results indicate that there was considerable difference between the two mixers. With mixer No. 1, uniform cement distribution throughout the batch was obtained only for the maximum mixing period of 135 sec. For all shorter mixing periods the cement contents of the samples from the front end of the mixer were appreciably greater than for the samples from the middle and rear of the mixer.

For mixer No. 2, the cement contents were comparatively uniform within the test batches for all except the minimum mixing period. In this latter case, the sample from the front end of the mixer had appreciably more cement than the samples from the middle and rear of the mixer.

Although large variations in cement content were obtained for different test samples, results of duplicate tests made on samples selected from the same mortar batches were very consistent. As an additional check, retests were made on two mortar samples which had the highest and lowest cement contents. Results of these check tests were:

Sample	Cement Content, per cent	
	Original	Retest
No. 1–10 B1	19.90	20.85
B2	19.28	20.70
Average	19.59	20.78
No. 2–36 A1	40.12	41.45
A2	39.42	39.85
Average	39.77	40.65

These check tests showed good reproducibility and indicate that the variations were not due to procedure in obtaining the test sample from the dried mortar sample.

Water-Cement Ratio.—The water-cement ratios were computed from the water-content and cement-content determinations on the mortar samples. Since these values were based on the dried mortar sample, they do not represent the true water-cement ratios of the concrete. However, there is a consistent relationship between the indicated and true values of water-cement ratio, and the data are suitable for determining the uniformity of the water-cement ratios for the samples from the different portions of the batch.

The project specifications required that the water-cement ratios of the concrete from the front, middle, and rear of the mixer not vary more than 10 per cent. On this basis, the data indicate that mixer No. 1 provided satisfactory mixing only when the 135 sec mixing time was used, and the shorter mixing periods were not satisfactory. For mixer No. 2, satisfactory uniformity in water-cement ratio was obtained when the mixing period was reduced to a low of 75 sec, although the variation in water-cement ratio exceeded the 10 per cent limit somewhat for the 105-sec mixing period. The 45-sec mixing period did not provide satisfactory uniformity of water-cement ratio.

In all instances, the variations in water-cement ratios of the concrete samples from the middle and rear of the batches were very small, and the large variations were between these values and those obtained for concrete samples from the front end of the mixer. Since satisfactory uniformity in water content was obtained for all but the 45-sec mixing period, it is evident that the unsatisfactory uniformity in water-cement ratios reflect the large variations in cement content.

Sand-Cement Ratio.—Sand-cement ratios were computed from the cement-content data, the weight of sand in the dry mortar sample being equal to the weight of the sample less the weight of cement. These sand-cement ratios were used to determine whether the variations between the samples from the front, center, and rear of the mixers were within the 10-per cent limit specified in the project specification. The data indicate that mixer No. 1 provided satisfactory mixing uniformity only for the maximum mixing period of 135 sec. Large variations in sand-cement ratios were obtained with the shorter mixing periods. The data are somewhat inconsistent for mixer No. 2 in that satisfactory mixing uniformity is indicated for the 90- and 75-sec mixing periods, with borderline uniformity for the 135-sec mixing period and unsatisfactory uniformity for the 105- and 45-sec mixing period. Where large variations in the sand-cement ratios within the concrete batches occurred, the variations were between the front portion and the remainder of the batch. These results also show the influence of the large variations obtained in the cement-content tests.

Strength-Cement Ratio.—Examination of the compressive strength and cement-content data indicated that there was a similar trend in variation for the different mixing conditions. Although these tests were made on samples taken from different batches representing the same mixing conditions, a comparison of the data was believed desirable. The strength - cement content ratios were computed for this purpose. Since cement content is on a percentage basis, these factors have no specific value in terms of strength per unit of cement, but serve only to compare the uniformity of the results.

Although individually, the compressive strengths and cement contents showed large variations within batches for mixer No. 1, the variations were comparatively small when considered in terms of strength per unit of cement. These data show that there is a consistent trend in the variation in compressive strength and in cement content and indicate that mixer No. 1 did not provide satisfactory mixing of the concrete when the mixing time was reduced from the maximum used.

TABLE II.—MIXER PERFORMANCE EVALUATION.

Test	Mixer No. 1				Mixer No. 2				
	Mixing Time, sec				Mixing Time, sec				
	135	105	75	45	135	105	90	75	45
Slump	*S*	*S*	*S*	*S*	*S*	*S*	*B*	*S*	*S*
Air content:									
Concrete	*S*	*S*	*S*	*U*	*S*	*S*	*S*	*S*	*S*
Mortar	*S*	*S*	*S*	*U*	*S*	*S*	*S*	*S*	*S*
Unit weight:									
Concrete	*S*	*S*	*S*	*S*	*S*	*S*	*U*	*S*	*S*
Mortar	*S*	*S*	*S*	*S*	*S*	*S*	*S*	*S*	*U*
Coarse aggregate, per cent	*S*	*S*	*S*	*S*	*S*	*S*	*S*	*S*	*U*
Water content	*S*	*S*	*S*	*U*	*S*	*S*	*S*	*S*	*U*
Cement content	*S*	*U*	*U*	*U*	*S*	*U*	*S*	*S*	*U*
Strength	*S*	*U*	*U*	*U*	*S*	*S*	*S*	*S*	*S*
Water - cement ratio	*S*	*U*	*U*	*U*	*S*	*U*	*S*	*S*	*U*
Sand - cement ratio	*S*	*U*	*U*	*U*	*B*	*U*	*S*	*S*	*U*
Performance	*S*	*U*	*U*	*U*	*S*	*U*	*S*	*S*	*U*

S = satisfactory
U = unsatisfactory
B = borderline

Evaluation of Mixer Performance:

In order to facilitate the evaluation of the performance of the mixers, performance ratings for the mixers in the various tests were assigned in accordance with the tentative limits selected for the tests. A summary of the performance ratings is presented in Table II. This table also shows the over - all performance ratings of the mixers for the different mixing periods used, based on performance in all tests conducted. The conclusions regarding mixer performance are based on these over-all performance ratings.

The results indicated that mixer No. 1 gave satisfactory mixing uniformity for the 135-sec mixing period and that mixing periods of 105 sec or less were not satisfactory. The performance of mixer No. 2 was somewhat inconsistent. Satisfactory performance is indicated for the 135-, 90-, and 75-sec mixing periods. The performance of this mixer was unsatisfactory for the 105- and 45-sec mixing periods.

Evaluation of the performance ratings in accordance with present Corps of Engineers requirements, which limits variations in sand-cement and water-cement ratios to 10 per cent, indicates the same order of mixer performance as when the evaluation was based on all tests conducted.

For evaluation of mixer performance in accordance with the Bureau of Reclamation limits for uniformity of mixing, the requirements are that the concrete from the front and rear of the mixer shall not vary from the average value more than 5 per cent in weight of coarse aggregate nor more than 0.8 per cent in unit weight of air-free mortar. On this basis, both mixers showed satisfactory performance for all mixing periods used.

No explanation has been found for the difference in performance of the two mixers, which were of identical construction. Project personnel stated that the mixers had been completely overhauled about one month before the tests were made and that the mixers were in excellent condition. New mixing and discharge blades had been installed in mixer No. 2, but only the mixer blades had been replaced with new ones in mixer No. 1. It is possible that this may have contributed to the difference in mixing action.

The arrangement for charging of the dry material into the mixers also may have contributed to the differences in mixer performance. Since the mixers were charged in opposite directions from the same batching hopper, the order of entrance of the materials into the mixers would differ. If cement entered first into the front end of the mixer, it is possible that it would not be properly distributed within the batch during the shorter mixing periods. Such a condition probably could be corrected by adjusting the order of charging the materials into the mixer.

Review of Mixer Performance Tests:

There are two basic requirements for a procedure for evaluating mixer performance: (1) the tests must provide an accurate measure of the uniformity of the concrete mixture throughout the batch; and (2) the test results must be available within a short period of time, preferably within a few hours, but not longer than 24 hr. Although all of the tests made gave some indication of the variations in the properties of the concrete within the batch, they were not all equally suitable for evaluating mixer performance. Comments on the suitability of the test procedure used follow:

Sampling.—Concrete samples for evaluating mixer performance should be obtained from the front, middle, and rear of the batch. For nontilting mixers, the samples may be obtained from the mixer or after the different portions of the batch have been discharged into separate containers. For tilting mixers, the samples should be obtained directly from the mixer so that there will be no question regarding the portion of the batch represented by the sample.

Slump.—For low-slump concrete, the slump variations within the batch may be of the same order as the variations due to the test procedure. This test is not sufficiently accurate for measuring uniformity of the concrete within the batch and is not a good mixer performance test.

Air Content.—Air content of air-entrained concrete may be determined on the concrete sample, or on the mortar portion of the concrete sample. The mortar test is preferred, as this is not influenced by variations in coarse aggregate content. The indications are that the maximum limit for variation in air content should be about 20 per cent.

Unit Weight of Concrete.—Unit weight is influenced to a large extent by the air content of the concrete. This test is not considered satisfactory for evaluating mixer performance.

Air-Free Unit Weight of Mortar.—For this test, the effects of air content and coarse aggregate content are eliminated. It is believed that this test provides a good measure of the distribution of the cement, water, and fine aggregate in the concrete mix. Based on the Bureau of Reclamation limit of 0.8 per cent for variation from the average value, a maximum limit of 1.5 per cent for variation between samples from the front, middle, and rear of the mixer is suggested for use.

Percentage of Coarse Aggregate.—This test provides a measure of the distribution of the coarse aggregate in the concrete. Care must be taken to obtain representative samples of the concrete. A maximum limit of 8 per cent, for variation within the batch is considered satisfactory.

Compressive Strength.—Although the strength test is a satisfactory measure of variation in the concrete, the time required for obtaining the test results makes it impractical to use this test for evaluation of mixer performance. An early age strength test would be subject to large variations and is not considered satisfactory.

Cement Content of Mortar.—Accurate determination of cement content will provide a good measure of mixing efficiency. For this purpose the cement content should be determined on a weight basis as a percentage of the weight of the mortar sample. Close control of the preparation and testing of the samples is necessary, and there is some question whether this test can be made properly in the field. Additional data are necessary for establishing a test limit for evaluating mixer performance.

Water Content of Mortar.—This test provides a satisfactory measure of the distribution of the water in the concrete batch. Additional data are necessary for establishing a suitable test limit for evaluating mixer performance.

CONCLUSIONS

Performance of Mixers:

The following conclusions are indicated by the test results obtained in this study:

1. Mixer No. 1 required a net mixing time of about 135 sec for satisfactory mixing of the concrete used for the pavement construction at this project.

2. Satisfactory mixing of the concrete was obtained with mixer No. 2 with a minimum mixing time of about 75 sec.

3. The revised mixing time requirements were satisfactory for the concrete mixers and mixing conditions used at this project.

4. Because of the variable results obtained and the lack of specific limits for evaluating mixer performance, the study did not provide an adequate basis for further modification of the mixing time requirements for the project on the basis of the mixer performance tests.

Mixer Performance Tests:

The study indicates that field testing of concrete mixers is practicable, and development of a standard test to evaluate mixer performance is considered desirable. The most suitable tests for evaluating mixer performance are:

1. Percentage of coarse aggregate in the concrete;
2. Air content of mortar;
3. Air-free unit weight of mortar;
4. Water content of mortar, and
5. Cement content of mortar.

These tests will provide a complete record of the distribution of the concrete materials in the batch.

Tentative limits for variation in test results have been indicated. However, these tests were limited to one concrete mix and one type of mixer. Additional tests with different types and sizes of mixers and different concrete mixes will be necessary for establishing suitable limits for a mixer performance test.

Acknowledgments:

The tests were conducted at the Clinton County Air Force Base, Ohio, by personnel of the Ohio River Division Laboratories. The work was done under authority of the Chief of Engineers, with the cooperation of the District En-

gineer, Huntington, W. Va., in making general arrangements for the test program. The cooperation of the contractor for the pavement construction, D. W. Winkleman Co., Inc., in adjusting his concrete mixing operations and in the handling of the concrete to provide the test samples is gratefully acknowledged.

DISCUSSION

Mr. Bryant Mather.[1]—I have two brief questions that I think might clarify matters.

Were the two mixers manufactured to be identical?

Mr. I. Narrow (*author*).—They were intended to be identical.

Mr. Mather.—Would you conclude that they performed differently because of real differences in the mixers themselves or might this have to do with unrevealed differences in the way they were batched?

Mr. Narrow.—I believe the differences were due to manner of batching, or possibly differences in the actual condition of the mixer. In either case they could have been readily overcome. Adjust the batching arrangements or the mixers themselves. No attempt was made to during the testing program. Our particular tests that indicated these wide differences were compressive strength and cement content, and in both of these tests the data were not available until some considerable period after the field study was made. This was also the reason we did not check into the actual reason for the difference in the mixers.

Mr. Mather.—The conclusion you would then reach perhaps is that these performance data could not be carried from one mixer to another simply because they were of the same manufacture and size.

One further question. You remarked that compressive strength would be a good test, but that the time required was extensive. Would it not perhaps be adequate to use early strength tests—for example, 3 days?

Mr. Narrow.—I believe that the possible variations you would get in the test results themselves would be an important factor here.

Mr. Mather.—I was thinking merely from a relative standpoint that an early strength test might suffice. In other words, the gain of time might be of greater benefit than the loss of quality in the test employed.

Mr. Narrow.—I would say that a mixer performance test should include such tests as could be made in a very short period of time; preferably within 24 hr. If the test is going to be used for the determination of the mixer in the field, then you should have your answer within 24 hr. If you are engaged in a general investigation, the time element is not that critical and therefore many other tests could be used.

Mr. Mather.—Are you not normally in the situation where you have a specified mixing time, and the question is: is it justified to reduce it? Can you not run safely at the less economical longer mixing time for a week before you find out whether it is safe to permit the shorter more economical time?

Mr. Narrow.—Well, this gets into another field, the question of the specification requirement itself. I do not think it is necessary to comment on that. If people feel they need a concrete strength test, then certainly they can make a concrete strength test. This is something for ASTM committee consideration.

---

[1] Engineer (Concrete Research), Concrete Division, U. S. Army Engineers Waterways Experiment Station, Jackson, Miss.

MR. T. B. KENNEDY.[2]—I would like to comment on what Mr. Mather said about using 3-day strength as an indication of uniformity of mixing.

I believe the standard deviation between cylinders varies less with age than does the average strength level. The coefficient of variation improves with age. For this reason 28-day results would be more reliable than 3-day results for indicating uniformity of mixing, but of course there is a great loss of time if one must wait for 28-day results. Three-day results can be used but they are not as reliable as 28-day ones.

Strength tests are not very practical to indicate mixer performance because of the time element, but in the case under discussion I think it was fortunate that they were made, because they nicely confirm results of the centrifuge tests, thus dispelling doubts as to the validity of the centrifuge results.

---

[2] Chief, Concrete Division, U. S. Army Engineer Waterways Experiment Station, Jackson, Miss.

INFLUENCE OF PHYSICAL CHARACTERISTICS OF AGGREGATES ON FROST RESISTANCE OF CONCRETE*

By George Verbeck[1] and Robert Landgren[2]

Synopsis

The need for an understanding of the influence of aggregates on the resistance of concrete to freezing and thawing is becoming increasingly important. The durability of concretes made with different aggregates depends upon the rate at which the aggregates become critically saturated in the concrete and upon the different physical responses of the aggregates to freezing.

An aggregate in concrete for which significant summertime drying is presumed can give satisfactory performance if the length of time required for the aggregate to become critically saturated is longer than its winter's exposure to wetting and freezing even though the aggregate would cause disruption if frozen while saturated. The time requirements for saturation of aggregates in concrete are analyzed in terms of the physical characteristics of the aggregates and the concrete. Important factors are the porosity and pore size distribution of the aggregate and the permeability and thickness of the mortar cover protecting the aggregates from water.

The different responses of aggregates to freezing when saturated depend upon the pore characteristics of the aggregates and the cement paste. Saturated aggregates of low porosity may accommodate pore water freezing by simple elastic expansion. Saturated aggregates of moderate or high porosity may fail internally because the particle dimension exceeds a certain critical size or may cause failure in the paste immediately adjacent to the aggregate particle because of aggregate pore water displacement. The magnitude of the hydraulic pressures developed is significantly influenced by the size of the aggregate particle and the permeability and air content of the surrounding paste.

The applicability of certain types of laboratory tests of the frost resistance of aggregates and concretes is questioned on the basis of these mechanisms.

A better understanding of the influence of aggregates on the resistance of concrete to freezing and thawing and deicer scaling is becoming increasingly essential. Sources of aggregates of known satisfactory performance are being depleted, particularly near some large metropolitan areas, and some so-called "marginal" aggregates are being rejected for use in concrete. In areas new to the use of concrete in pavements, performance records are not available for the local aggregates.

It is believed that some of the so-called "marginal" aggregates are rejected for use in concrete because of inappropriate testing techniques and that other aggregates now considered as marginal or unacceptable can be successfully used provided certain simple and frequently attainable requirements are imposed upon the aggregate and the concrete.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Manager, Applied Research Section, Research and Development Laboratories, Portland Cement Assn.

[2] Associate Research Engineer, Applied Research Section, Research and Development Laboratories, Portland Cement Assn.

It is the purpose of this paper to consider the mechanisms by which the pores in aggregates influence concrete durability, to assist in providing a fundamental and rational basis for proper evaluation of aggregates, and to establish some principles whereby aggregates of moderate frost susceptibility may be successfully used in concrete. To do this requires an understanding both of the mechanisms involved and of the influence of the various physical characteristics of the aggregates on these mechanisms and the resulting performance of the concrete.

Special cases in which concrete durability may be directly influenced by chemical reactions of the aggregate or possibly by differences in the thermal expansivities of the components of the concrete will not be considered.

THE PHYSICAL MECHANISMS

The general problem of the resistance of concrete to freezing and thawing and the related action of de-icing chemicals has been presented in numerous publications (**1–8**).[3] The effectiveness of air entrainment in providing a high order of durability to concretes made with good quality aggregates has been adequately demonstrated.

The general concepts that have successfully provided an understanding of the frost resistance of cement paste (**3**) are also applicable to aggregates. Aggregates may cause disruption of concrete by the generation of high destructive hydraulic pressures during freezing. By analogy with paste, the magnitude of the hydraulic pressure developed in aggregates depends upon their degree of saturation (proportion of total void space filled with water) and the permeability and size of the aggregate particle. Furthermore, if the degree of saturation of the aggregate is sufficiently high (above the "critical," 91.7 per cent) so that the remaining air-filled void space cannot accommodate the 9 per cent expansion of water during freezing, then water will be expelled into the paste surrounding the aggregate particle and potentially destructive hydraulic pressures may develop there as well as in the aggregate. Thus, the properties of paste, its permeability, air content, and porosity, are also involved in the problem of aggregate durability. In addition, the paste can significantly influence the degree of saturation of the aggregate in concrete by limiting the ingress of water; the protection afforded in this manner depends upon the permeability and thickness of the paste or mortar cover separating the aggregate from the wet surface of the concrete.

A more detailed consideration of the various mechanisms by which the various physical characteristics of the aggregate and of the paste can influence the durability of the concrete reveals that the effects of some of these characteristics are closely interrelated and that some may have several and perhaps opposing effects.

Analysis of the mechanisms is considerably simplified by separating the over-all problem into two distinct aspects within which the individual roles of the pertinent physical characteristics of the aggregate and the paste can be developed more clearly. These major aspects and the important factors involved are as follows:

1. The time required for an aggregate to become critically saturated when in concrete exposed to water as influenced by:

(*a*) Pore size and porosity of aggregate.

---

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

(*b*) Thickness and permeability of protective mortar cover.

2. The various phenomena in the freezing of fully saturated aggregate demonstrating:

(*a*) Elastic accommodation by aggregate.

(*b*) "Critical" size of aggregate (internal hydraulic pressures).

(*c*) Influence of confining mortar (external expulsion distance and external hydraulic pressures).

(*d*) The influence of various factors modifying these effects of freezing, that is, soluble materials, degree of saturation and freezing point depression in fine aggregate pores.

These various phenomena in freezing as influenced by the physical properties of aggregate and paste will now be considered in detail and supporting experimental data obtained by this laboratory presented where appropriate.

Time Required for Critical Saturation:

Most concrete in above-ground structures or in pavements placed on free draining subbases does not normally remain saturated but instead is subjected to drying, particularly during the summer. A single drying period occurring some time prior to freezing produces a marked improvement in concrete durability as has been demonstrated in numerous laboratory tests. Therefore for most purposes, we must be concerned not only about the durability of the concrete were it to become critically saturated but more importantly about the length of time required for concrete to attain such a state of vulnerability to freezing when exposed to the wetting-and-freezing conditions of winter. Following the beneficial drying of summer, a few aggregates may require only a few weeks of wetting to become critically and damagingly saturated. Most aggregates, because of their porosity and pore size or because they are used in concretes of low permeability (low water-cement ratio), may require many months to attain a critical saturation and hence successfully pass through the freezing season to the next drying and recuperating season.

Consequently, from this realistic point of view, legitimate arguments have been made against indiscriminate use of test data obtained by freezing of continually wetted concretes made with presaturated aggregates as the sole criterion for aggregate acceptability (6). On the basis of unrealistic tests, an aggregate may be condemned as non-durable when, in fact, it might give satisfactory performance in structures above ground or in pavements which are on free draining subbases and are exposed to summertime drying.

The two major factors that appear to determine the time required for an aggregate in concrete to become critically saturated are: (*a*) the nature of the aggregate—that is, the porosity and the size distribution of pores in the aggregate, and (*b*) the nature of the mortar surrounding, and in a very real sense protecting, the aggregate in the concrete from the supply of water external to the concrete—its permeability and its thickness, that is, the distance from a piece of aggregate to the surface of the concrete.

These factors, currently under study, are not as yet understood sufficiently well to permit an exact quantitative calculation of their individual influences; however, present understanding is sufficient to permit a qualitative and semiquantitative interpretation of their influences.

Pore Size and Porosity:

These characteristics of aggregates in relation to the durability of concrete have been studied by many investigators. Rhoades and Mielenz (9) compared aggregates having pores of small or large diameter when cast in similar cement pastes and concluded that the aggregate with the smaller pores would attain and retain a higher degree of saturation and hence be most likely to fail when frozen. Blanks (2) reported tests for capillary water retention of aggregates and in addition showed that certain aggregates had pores that were small enough to compete successfully with some hydrated cement pastes for available pore water.

In order to consider this problem in greater detail we will use, as a simple working model, a system consisting of a piece of dry aggregate, of known porosity and pore size, separated from a supply of free water by a membrane of mortar of known thickness and permeability. This model simulates a wetted concrete pavement surface with a piece of dry aggregate a known distance beneath the surface. For convenience we will assume that the mortar membrane is saturated and serves only to transmit water from the surface of the concrete into the aggregate. The rate at which water moves through this mortar membrane depends upon the characteristics of the membrane and a "pressure" difference resulting from capillary tension across the membrane. The "pressure" difference, ΔP, is approximately expressed by the following relationship (10):

$$\Delta P = \frac{d \times R \times T}{M} \ln \frac{\% \, RH}{100}$$

where:

d = density of water at temperature T, g per cu cm,

R = gas constant = $82.05 \dfrac{\text{cu cm} \times \text{atmos}}{\text{mole} \times \text{deg K}}$,

T = absolute temperature, deg K,

M = molecular weight of water = 18.02, and

$\% \, RH$ = relative humidity in aggregate, per cent.

This relationship indicates that the rate of water movement into the aggregate under the postulated conditions depends significantly upon the relative humidity within the aggregate and is reduced rapidly as the relative humidity within the aggregate approaches 100 per cent. For example, the pressure gradient causing water movement into aggregate at 90 per cent relative humidity is only 15 per cent of that for aggregate at 50 per cent relative humidity.

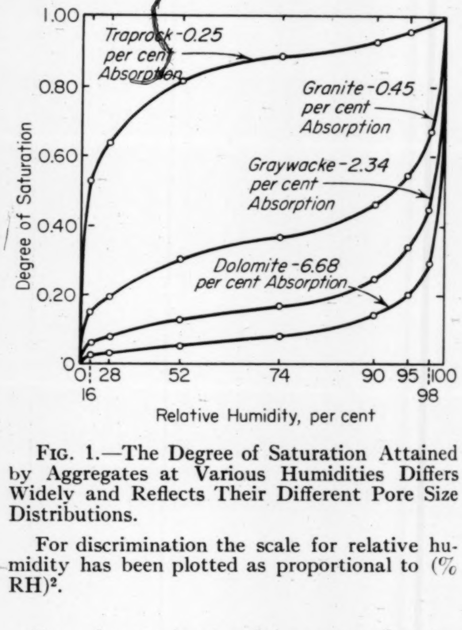


FIG. 1.—The Degree of Saturation Attained by Aggregates at Various Humidities Differs Widely and Reflects Their Different Pore Size Distributions.

For discrimination the scale for relative humidity has been plotted as proportional to $(\% \, RH)^2$.

In order to express these rates of water movement in terms of degree of saturation of an aggregate, it is necessary to know the water vapor adsorption characteristics of the aggregate. As shown in Fig. 1, the relationships between degree of saturation and relative humidity differ significantly among aggregates; aggregates with fine pores such as the traprock adsorb considerable water vapor and attain a higher degree of saturation at any particular humidity than aggregates with a coarse pore system such as the dolomite.

From these relationships and from the logarithmic dependence of rate of water movement into the aggregate on the relative humidity within the aggregate, it is apparent that an aggregate with a fine pore structure similar to the traprock would reach a high degree of saturation much more rapidly than an aggregate with the coarse pore structure of the dolomite, even if the aggregates had the same porosity.

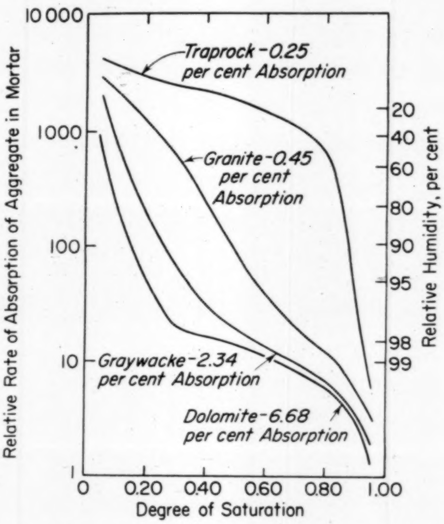


FIG. 2.—Aggregates Encased in Mortar Absorb Water at Different Rates Because of Their Different Pore Size Distributions.

Note the logarithmic dependence of this absorption rate upon the relative humidity in the aggregate at various degrees of saturation.

Moreover, as is also shown in Fig. 1, these particular aggregates differ not only in pore size distribution but also in total porosity or absorption. These porosity differences serve further to accentuate the differences in behavior of these aggregates. For example, at 52 per cent relative humidity both the traprock and dolomite aggregates would be subject to equal "pressures" and hence have equal rates of water uptake, but the traprock would already be 82 per cent saturated and would require only 0.045 per cent by weight of additional water for total saturation, whereas the dolomite would be only 6 per cent saturated and would require 6.28 per cent by weight of additional water for total saturation.

Figure 2 shows the rates at which these aggregates tend to absorb water at various stages of saturation and at the various relative humidities. It may readily be observed that the traprock has a much higher rate of absorption than the dolomite, even when the aggregates are compared at equal degrees of saturation.

From the above, it should be apparent that for aggregates with similar pore size distributions, the one with high porosity should require more time to attain any particular degree of saturation than the one of low porosity. For example, for two such aggregates, one of 15 per cent porosity would require three times as long to increase in internal humidity from 50 to 51 per cent than would one of 5 per cent porosity. This is a matter of very practical importance.

With any aggregate containing coarse pores and a sufficiently thick mortar cover of low permeability, the time required for critical saturation will have considerable significance. If the time required to attain a disruptive state of saturation (assume 96 per cent) were 3 months for the aggregate having 5 per cent porosity, the aggregate with 15 per cent porosity would require 9 months. This additional 6-month period would be more than sufficient to take concrete with the 15 per cent porosity aggregate through the critical wetting-and-freezing season and satisfactory performance would be expected.

Hence it may be seen that under some circumstances it may be beneficial to the performance of concrete for the aggregate to have a high porosity, and that aggregates of some intermediate porosity in concrete may actually show the poorest performance.

These considerations of the time required for critical saturation have qualitative practical significance. A complete quantitative evaluation requires additional information about uniformity of distribution of absorbed water in aggregates, the process of drying of aggregates in concrete, and the influence of the mortar other than that of transmitting water directly to the aggregate on the rate of saturation of the aggregate.

TABLE I.—TIME REQUIRED FOR DESTRUCTIVE SATURATION OF AGGREGATE DEPENDS UPON THE PERMEABILITY AND THICKNESS OF THE PROTECTIVE MORTAR COVER.

Mortar Cover		Days of Wetting Sustained Before Failure (Popout) at Various Thicknesses of Mortar Cover		
Water-Cement Ratio by Weight	Permeability, K_1, cm per sec	⅛-in. Thick	¼-in. Thick	⅜-in. Thick
0.70	3000×10^{-9}	111	879	792
0.45	1	477	885	980

Thickness and Permeability of Mortar Cover:

It is reasonable to assume that the mortar cover transmits water by capillary tension in a manner approximating Darcy's law for flow under hydrostatic pressure and therefore that the rate of flow would decrease as the thickness of the membrane increases, and that a highly permeable membrane would transmit water more rapidly than a membrane of low permeability.

The permeability of paste or mortar depends significantly upon the water-cement ratio and the degree of hydration of the cement. For well hydrated pastes the permeability may be increased by as much as 100 fold as the water-cement ratio is increased from 0.40 to 0.70 by weight (11). Such differences in permeability have a significant influence on the rate at which the mortar membrane will transmit water into an aggregate.

That the permeability and thickness of the mortar cover does significantly affect the time required for an aggregate to become destructively saturated in concrete is demonstrated in Table I. These data, which are typical of results of our various laboratory evaluations, were obtained by embedding 1-in. cubes of a dolomite aggregate (6.68 per cent absorption) to the depths shown in mortar slabs having water-cement ratios of 0.70 and 0.45 by weight. After a preliminary 28 days of moist curing, the slabs were air dried to constant weight at 73 F and 50 per cent relative humidity and then subjected to wetting by continuous ponding of water on the upper surface. The slabs were subjected to freezing every third day (sides and bottom insulated) to determine whether any of the aggregate cubes had become destructively saturated as evidenced by the occurrence of a popout. It may be seen that the length of time required to destructively saturate the aggregate was beneficially lengthened by the use of the low water-cement ratio mortar and a greater thickness of cover.

In field concrete, the use of low water-cement ratios could extend the time required for critical saturation of aggregate sufficiently to provide satisfactory performance even though the aggregate would have marginal or poor performance in air-entrained concrete of high water-cement ratio.

In the case of concrete pavements, loss of protective mortar by wear or by grinding (to meet surface smoothness tolerances) may alter the performance of aggregate particles near the concrete surface.

The Various Phenomena in the Freezing Saturated Aggregate:

In addition to the effect on the length of time the concrete can withstand exposure to water before the aggregate becomes critically saturated and potentially vulnerable to freezing action, the pore characteristics of aggregates and the mortar in concrete significantly influence the response of the critically saturated aggregate to freezing.

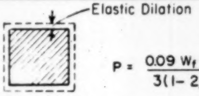
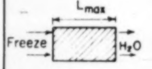
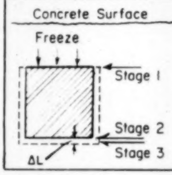
	A Quartzite Absorption = 0.1 per cent	A Chert Absorption = 2.1 per cent $K_1 = 1 \times 10^{-10}$ cm per sec	A Dolomite Absorption = 6.7 per cent $K_1 = 300 \times 10^{-10}$ cm per sec
Elastic Accommodation – Instantaneous Freezing			
 $P = \dfrac{0.09\, W_f\, E}{3(1-2\mu)}$	P = 780 psi (Accommodated)	P = 17,000 psi (Fails)	P = 35,000 psi (Fails)
Critical Size – Unconfined Freezing at 1.5 in. per hr			
 $L_{max} = \dfrac{27.7\, K_1\, T}{0.09\, dW_f/dt}$	(No Damage)	L_{max} = 0.5 in. (Fails if Larger)	L_{max} = 3.3 in. (No Damage)
Required Expulsion Distance – Freezing While Confined			
 Concrete Surface Freeze Stage 1 Stage 2 Stage 3 ΔL $\Delta L = \dfrac{0.09\, W_f\, L}{5A}$	(No Damage)	Cube Size, in. — ΔL, in. 1/4 — 0.0015 1/2 — 0.0030 1 — 0.0060	Cube Size, in. — ΔL, in. 1/4 — 0.0040 1/2 — 0.0080 1 — 0.0160

FIG. 3.—Summary of Various Effects of Freezing Saturated Aggregates.

The influence of pore characteristics of aggregates and mortar on the reaction of saturated aggregate when frozen under various conditions is summarized in Fig. 3. Three different types of aggregates have been used to demonstrate three different responses to freezing, depending upon the pore characteristics of the aggregate as measured by total porosity (absorption) and permeability.

The different types of aggregates typifying the different types of response on freezing are:

(*a*) An aggregate of *low porosity*, about 0.3 per cent by volume, and perhaps typical of some quartzites, marbles, or traprocks. Such an aggregate will demonstrate *elastic accommodation*.

(*b*) An aggregate of *moderate or high porosity* but of *low permeability*, perhaps typical of cherts and other absorptive aggregates with a fine pore structure, which can cause failure because of *high internal pressures*, i.e., *critical size effects*.

(*c*) An aggregate of *moderate or high porosity* but of relatively *high permeability*, perhaps typical of many limestones, dolomites, sandstones, and other absorptive aggregates with a relatively coarse pore structure which can cause failure because of *high external pressures* in the surrounding mortar.

Elastic Accommodation:

Under conditions of normal use, the concrete is not subjected to extremely rapid freezing, and therefore a significant portion of the excess volume of water created in the freezing aggregate has time to escape and thus keep internal pressures below the tensile strength of the aggregate. It is not necessary that all of the excess volume be expelled in order to avoid rupture of the aggregate as the aggregate can elastically accommodate a portion of this excess volume without rupture.

The principle of elastic accommodations is clearly revealed by considering the limiting case of instantaneous freezing of completely saturated aggregate. Under such circumstances, none of the water within the particle has time to escape or be expelled. For avoidance of failure, all of the volume increase which accompanies the formation of ice from liquid water (9 per cent of the water volume) must be accommodated elastically within the aggregate.

The ice pressure, or its counterpart the tensile stress which is created within an aggregate, can be estimated from the relation:

$$P = \frac{0.09\ W_f E}{3(1 - 2\mu)}$$

where:

P = internal ice pressure or aggregate tensile stress required to increase volume of aggregate sufficiently to accommodate expansion, psi,

W_f = volumetric fraction of freezable water in aggregate, cu cm per cu cm = aggregate porosity for saturated aggregate, and

E = modulus of elasticity of aggregate, psi, and

μ = Poisson's ratio of aggregate.

It may be noted in Fig. 3 that the calculated tensile stress created on instantaneous freezing of a saturated aggregate having an absorption of 0.1 per cent by weight (porosity = 0.3 per cent) is only about 780 psi, a stress capable of being withstood by this aggregate. There is a class of aggregates, those that have porosities up to about 0.30 per cent by volume, that are completely invulnerable to freezing action and can be frozen instantaneously even when completely saturated.

Obviously, saturated aggregates containing significantly greater amounts of freezable water would require correspondingly greater dilations in order to accommodate the internal volume increase of the water on freezing. Estimates of the tensile stresses corresponding to these required dilations are shown in Fig. 3 for the aggregates having 2.1 and 6.7 per cent absorption by weight. It is seen that the required tensile stresses are much beyond the tensile strengths of these aggregates and hence that rupture would be expected.

Experimental verification of elastic accommodation during very rapid freezing of saturated aggregates having porosities of about 0.3 per cent by volume (0.1 per cent absorption by weight) or less is shown in Table II. For such aggregates, vacuum saturated and rapidly frozen at 6 F per sec, the calculated tensile stresses

TABLE II.—FULLY SATURATED AGGREGATES OF LOW POROSITY DO NOT FAIL WHEN FROZEN RAPIDLY BECAUSE OF INTERNAL ELASTIC ACCOMMODATION.

1 by ⅜ by ⅜-in. aggregate prisms frozen at 6 F per sec.

Aggregate	Absorption, per cent by weight	Modulus of Elasticity, E, psi	Calculated Stress, psi	Results of Freezing
Limestone	1.7	3×10^6	7600	Cracked
Sandstone	1.1	2	4400	Cracked
Limestone	0.7	5	5500	Cracked
Traprock	0.17	8	2000	Cracked
Limestone	0.10	6	1500	No distress
Marble	0.05	10	700	No distress

required for elastic accommodation were 1500 psi or less. Stresses of this magnitude are approximately equal to or below the tensile strength of these aggregates which withstood freezing under these most critical conditions of test. Aggregates with higher porosities and calculated tensile stresses greater than their tensile strength failed by rupture.

Of course, under conditions of normal use in concrete, aggregates are not subjected to such very rapid freezing, and a significant portion of the excess volume of water created in the aggregate may be expelled during freezing.

Unconfined Freezing and Critical Size of Aggregate:

The magnitude of the hydraulic pressure developed in a saturated aggregate particle during freezing depends upon the rate of freezing and the porosity, permeability, and size of the aggregate particle (**12**). If for simplicity it is assumed that both the propagation of freezing and the expulsion of water are unidirectional, the maximum hydraulic pressure can be estimated, using Darcy's law, as:

$$P_{\max} = \frac{0.09\, dW_f/dt\, L}{27.7K_1}$$

where:

$P_{\max}$ = maximum pressure, psi,
dW_f/dt = rate of freezing of water,
= aggregate porosity × rate of linear propagation of freezing zone, cm per sec,
L = dimension of aggregate in direction of freezing and expulsion of water, in.,
K_1 = permeability coefficient of aggregate, cm per sec, and
27.7 = conversion factor, in. hydraulic head per psi.

Since $P_{\max}$ cannot exceed the tensile strength of the aggregate, this equation can be used to estimate the maximum permissible size or "critical size" of the aggregate as follows:

$$L_{\max} = \frac{27.7\, K_1 T}{0.09\, dW_f/dt}$$

where:

$L_{\max}$ = maximum permissible size—"critical size," in., and
T = tensile strength of aggregate, psi.

The results of calculations based on this equation and assuming a freezing rate of 1.5 in. per hour (1.06×10^{-3} cm per sec) are shown in Fig. 3. This freezing rate is considered to approximate rates of freezing encountered in natural exposures and in some laboratory freezing tests. It should be noted that the chert type aggregate of moderate absorption, but of low permeability (fine pores), has a calculated critical size at this freezing rate of only 0.5 in. However, aggregates of high porosity can have a very large critical size if they also have a high permeability. For the dolomite aggregate shown in Fig. 3 and for the assumed rate of freezing, a critical dimension of approximately 33 in. is calculated. A saturated aggregate of this type should not fail by the development of high internal hydraulic pressure when unidirectionally frozen at the prescribed rate, provided the aggregate is unconfined and the excess volume of water created during the freezing can be expelled.

The preceding calculations were based on the assumption of unidirectional expulsion of water, an assumption which permits simple mathematical expression. During the actual unidirectional freezing, some of the excess volume of water would be expelled laterally and would tend to relieve the internal hydraulic pressure and hence increase the practical critical size of the aggregate. Complete mathematical expressions including the lateral expulsion of water have not been obtained.

The concept of critical size of aggregate can be demonstrated by laboratory tests such as those shown in Table III. These results were obtained by the freezing and thawing of various size fractions of a particular chert (3.6 per cent absorption) previously vacuum saturated. These data clearly show that large particles of this aggregate are much more vulnerable to freezing than small particles.

TABLE III.—LARGE PARTICLES OF A SATURATED CHERT AGGREGATE ARE MORE VULNERABLE TO FREEZING THAN SMALL PARTICLES.

	Size Range of Particles		
	1½ to ¾ in.	¾ to ⅜ in.	⅜ in. to No. 4
	Per cent passing ¾-in. sieve	Per cent passing ⅜-in. sieve	Per cent passing No. 4 sieve
Original	0	0	0
12 cycles, freezing and thawing	33	12	1
51 cycles, freezing and thawing	61	34	6

Chert aggregate, 3.6 per cent absorption by weight, "saturated" by 3 hours evacuation and 5 days under water. Freezing and thawing under water.

Freezing While Confined by Mortar:

To avoid failure during freezing, a saturated aggregate particle having an absorption greater than that which can be elastically accommodated (about 0.1 per cent) and a size smaller than its critical size must be able to expel water into the surrounding air-entrained mortar without the development of hydraulic pressures that exceed the tensile strength of the concrete. The factors of primary importance to this mechanism are the freezing rate, aggregate size and porosity, and the permeability and air content of the surrounding paste.

Let us first consider the distance that the expelled water must move into the surrounding paste for volumetric accommodation. We will assume unidirectional freezing of an aggregate cube with the freezing zone entering one face and water being expelled from the other five faces. As a simplifying and reasonably justifiable condition, we will further assume that equal amounts of water are expelled from all five surfaces and that the expelled water completely fills the air voids for a uniform distance, ΔL, around the five sides of the cube. ΔL can be calculated as follows:

$$\Delta L = \frac{0.09\ W_f L}{5A}$$

where:

ΔL = distance required for volumetric accommodation of expelled water, in.,
L = dimension of aggregate cube, in.,
W_f = volumetric fraction of freezable water in aggregate, cu cm per cu cm,
= porosity of saturated aggregate, and,
A = air content of paste, fractional = 0.18 (estimated).

Figure 3 contains the results of calculations of expulsion distances for various size cubes of the chert and dolomite. It can be seen that the expulsion distances calculated on the above basis are in general relatively small. For small size particles of the chert having intermediate porosity, the calculated expulsion distances of 0.0015 and 0.0030 in. would appear to be sufficiently small to be accommodated by the paste without the development of distress, particularly when these distances are compared to the allowable distance, the bubble spacing factor, L, of 0.0100 in. (3) which is considered satisfactory to protect air-entrained concrete. For the 1-in. chert cube, the calculated distance of 0.0060 in. would also appear to be within this normally ascribed limit, but, as has been previously shown, failure of this particular size of chert could result solely from considerations of critical size if this aggregate were frozen at a rate of 1.5 in. per hour.

The expulsion distances calculated for the dolomite aggregate of high porosity are large, perhaps excessive for the larger sizes of the aggregate. The required expulsion distance depends significantly on the aggregate size and air content of the surrounding paste. Air entrainment can significantly reduce the required expulsion distance and hence should serve to moderate the high vulnerability of aggregate considered as unsound when used in non-air-entrained concrete.

TABLE IV.—TIME REQUIRED FOR DESTRUCTIVE SATURATION OF AGGREGATES IN MORTAR IS SIGNIFICANTLY LONGER FOR SMALLER SIZE AGGREGATE PARTICLES.

0.70 W/C mortar—⅛-in. mortar cover.

Aggregate	Absorption, per cent by weight	Days of Wetting Sustained Before Failure (Popout)		
		Aggregate Cube Size, in.		
		1	¾	½
Dolomite No. 1.....	6.68	111	213	>980
Dolomite No. 2.....	3.59	90	177	309

The advantageous effect of using a small maximum size of such aggregate in concrete on the required expulsion distance and potential destructive action appears obvious. Experimental laboratory verification of the beneficial effect of reduced aggregate size is shown in Table IV. These data were obtained in tests similar to those described in connection with Table I. It may be observed that the time required for destructive saturation is significantly lengthened by the use of smaller size of aggregate.

As previously discussed, the distances shown in Fig. 3 do not appear disruptively high when compared to calculated protected thicknesses of pastes of about 0.0100 in. in air-entrained concrete. However the magnitude of the hydraulic pressures associated with these distances is of interest. The hydraulic pressures developed at three important stages of the freezing are shown as stages 1, 2, and 3 in Fig. 3 as follows:

Stage 1.—The instant of freezing of the exterior face of the aggregate (that face closest to the freezing surface of the concrete). At this instant, the permeability of the aggregate, its total porosity and the freezing rate govern the pressures generated by movement of water through the aggregate into paste air voids present at the aggregate surfaces.

Stage 2.—The instant of freezing of the interior surface of the aggregate when pressures are determined by the rate at which water is ejected from the aggregate upon freezing, the depth of paste saturated by the aggregate (ΔL) and the paste permeability.

Stage 3.—The freezing of the paste plus air void system immediately adjacent to this interior surface which is completely saturated for a distance ΔL. This pressure is a function of the freezing rate, paste air content, and the permeability of the paste.

Accurate measurements can be made of the pertinent physical characteristics of aggregates, and reasonable assumptions can be made regarding rates of freezing of aggregates and the air content of the surrounding paste. However, the least known factor is the permeability of the paste, which has a very significant and direct effect on hydraulic pressure. This permeability depends upon the water-cement ratio of the paste, the degree of hydration of the cement, and apparently is greatly influenced by any prior drying of the paste.

Calculated hydraulic pressures at the

three different stages of freezing of the chert and dolomite aggregates as shown in Table V are based on the several possible paste permeabilities (which vary over 10,000 fold) rather than on a single assumed and perhaps misleading "representative" permeability. The calculations are based on directly measured aggregate characteristics and estimated factors for rate of freezing (1.5 in. per hr) and air content of paste (18 per cent).

TABLE V.—MAGNITUDE OF THE HYDRAULIC PRESSURES DEVELOPED AT THE VARIOUS STAGES OF FREEZING DEPENDS UPON THE PORE CHARACTERISTICS OF THE AGGREGATE AND THE PERMEABILITY OF THE SURROUNDING PASTE.

½ in. aggregate cubes, air content of paste = 18 per cent.

Paste Characteristics			Hydraulic Pressure Developed at Various Freezing Stages,[b] psi					
Water-Cement Ratio, by weight	Prior Treatment	Permeability of Mature Paste, K_1, cm per sec	For a Chert Permeability = 1×10^{-10} cm per sec $\Delta L = 0.003$ in.			For a Dolomite Permeability = 300×10^{-10} cm per sec $\Delta L = 0.008$ in.		
			Stage 1[a]	Stage 2	Stage 3	Stage 1[a]	Stage 2	Stage 3
0.30	Never dried	1×10^{-13}	1020	6100	18 600	8	43 000	49 500
0.50	Never dried	150	1020	41	200	8	290	700
0.70	Never dried	1200	1020	5	44	8	36	117
0.50	Dried to 79 per cent RH and resaturated	10 000	1020	1	4	8	4	11

[a] Unidirectional expulsion of water assumed.

[b] See Fig. 3.

The results shown in Table V clearly demonstrate that the permeability of the paste is a major factor influencing the hydraulic pressures developed. These data also indicate that the permeability of a mature paste of 0.50 water-cement ratio can be increased by as much as 60 to 70 fold by prior partial drying. Such changes can have very large and significant effects on the hydraulic pressures developed, and more information on the influence of drying on permeability is desirable.

It should be noted that whereas low paste permeabilities would create high hydraulic pressures during freezing of saturated aggregate, low permeability would also have an opposite and beneficial effect of decreasing the rate of saturation of the aggregate as previously discussed.

As shown in Table V the most damaging stage of freezing, at least that stage developing the highest hydraulic pressure, does not occur during the freezing of the aggregate itself but rather during the subsequent freezing of the completely saturated paste (paste plus air voids) adjacent to the aggregate.

The hydraulic pressures shown for the first stage of freezing of the two aggregates were calculated, assuming unidirectional expulsion of water. As previously discussed, the hydraulic pressures actually created during the first stage of freezing may be several fold less than the pressures shown because of lateral expulsion of water. However, the relative pressures calculated for the chert and dolomite materials should be correct; that is, the pressure created at the beginning of freezing of the low-permeability chert should be approximately 100 times greater than for the high-permeability dolomite.

It would appear that a saturated porous aggregate of low permeability would be more likely to cause "popouts" in a concrete surface than a saturated aggregate of the same porosity but of high permeability. In the former case, a high initial hydraulic pressure is created close to the freezing surface of the concrete, precisely at the location where the lowest pressure is required to cause rupture of the concrete and the occurrence of a "popout."

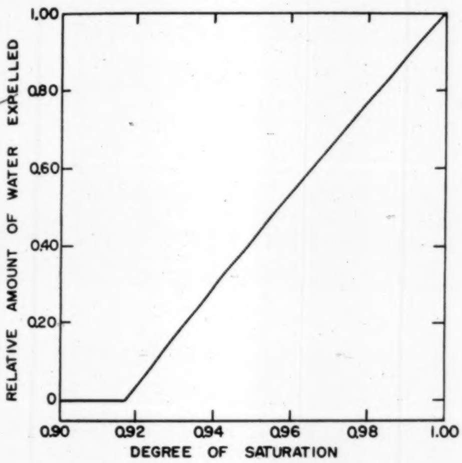


Fig. 4.—The Relative Amount of Water Expelled and Hence the Disruptive Effect of Freezing Depend Upon the Degree of Saturation of the Aggregate.

Modifying Factors:

In the preceding discussions of the various aspects of freezing of aggregates it has been assumed, for convenience and simplicity of presentation, that the various porous materials were completely saturated and that all of the water contained in the pores of the aggregate would freeze at the normal freezing point of water, 32 F.

In an actual case of freezing of an aggregate, neither of these assumptions is necessarily true. If the aggregate is not saturated, or if all of the water does not freeze, either because of the presence of soluble material or because of the fineness of aggregate pores, then the maximum calculated parameters for hydraulic pressure, expulsion distance, and critical size must all be modified by appropriate factors.

In most instances, the freezing of aggregates in concrete involves aggregates that are not totally saturated, total saturation being extremely difficult to attain. The degree to which total saturation is approached influences significantly the damaging effects of the freezing. The hydraulic pressure created during freezing is a direct function of the amount of water which must be expelled from the freezing zone. If the aggregate is totally saturated, the amount of water expelled is approximately 9 per cent of the total volume of water contained. If the porous system is only 91.7 per cent saturated, the remaining unfilled 8.3 per cent of pore space is sufficient to accommodate the 9 per cent expansion that accompanies the

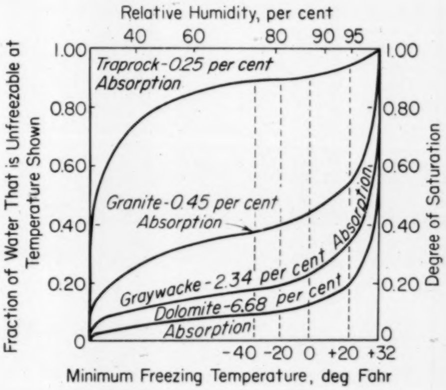


Fig. 5.—The Fraction of Pore Water Remaining Unfrozen at Various Temperatures Depends Upon the Size of the Aggregate Pores and Can Be Estimated from the Adsorption Isotherm.

For discrimination, the scale for relative humidity has been plotted as proportional to (% RH)^2.

formation of ice. Since no water need be expelled from the porous system, macroscopic hydraulic pressures are not created. Figure 4 shows the degree to which the hydraulic pressures and expulsion distances previously described are decreased in instances where the aggregate is less than totally saturated.

In addition, other factors modify the effects of freezing. Some of the water contained in the pores of an aggregate will not freeze at 32 F. Water-soluble salts and cement alkalies lower the freezing point of the pore liquid. The freezing point depression due to this effect varies with the materials present and their concentration but usually is not great enough to prevent freezing of the pore liquid at moderate freezing temperatures.

Depression of freezing point also occurs with water in fine capillaries or surface adsorbed. Powers and Brownyard (**13**) reviewed the mechanisms involved in these physical processes and calculated the resulting theoretical freezing point depression. Figure 5 shows such theoretical freezing point depressions, calculated as a function of equilibrium relative humidity, superimposed on the water adsorption isotherms previously presented. It is apparent from this figure that significant amounts of pore water will not freeze in certain aggregates. In the case of the traprock, only about 10 per cent of the water absorbed by vacuum saturation should freeze at −40 F. It should be noted, however, that those aggregates having higher total absorptions have the lower reductions in amount of freezable water due to the effects of capillarity and adsorption. Such reductions in freezable water result in smaller volumes of water being expelled and thereby moderate the disruptive actions.

DISCUSSION OF VARIOUS TEST METHODS

The actual field durability of concretes made with various aggregates depends upon the rate of saturation of the aggregates and their response to freezing. These two phenomena involve various physical characteristics of the aggregates —particle size, permeability, porosity, and pore size distribution—and of the mortar or paste component of the concrete—its air content, permeability and its thickness as protective cover. It has been shown that certain characteristics of the aggregates or mortar can play a dual role with resulting opposite effects on concrete performance. Aggregates of high porosity and mortars of low permeability beneficially reduce the rate of saturation of aggregates in concrete but also have deleterious effects during the freezing of saturated aggregates. For this reason, simple correlations or effects are not to be expected.

The mechanisms and mathematical expressions presented are applicable in a qualitative and semi-quantitative sense. The fundamental principles involved are sufficiently well defined to permit examination of some of the physical tests used to evaluate the so-called "durability" of aggregates in concrete.

The need for rapid and convenient tests for the "durability" of aggregates is apparent, and a number of test methods have been reported in the technical literature. Some of these tests yield information regarding the frost susceptibility of various aggregates, but the limitations of such information and the degree to which it may or may not apply to actual field conditions warrant consideration.

It would appear reasonable that a test method should incorporate the determination, or at least consideration, of those physical characteristics of aggregates actually involved in the mechanisms of saturation and response to freezing. In tests such as the sodium or magnesium sulfate soundness tests, the mechanism of disruption is different from that resulting from the freezing of water. Although results of this type of test must reflect something about the pore characteristics of the aggregates, the results cannot be directly and rationally interpreted in terms of any of the factors of importance to the actual mechanism of freezing in concrete. Such test results have only rough and uninterpretable empirical correlation with concrete performance.

A somewhat more realistic type of test involves the freezing of unconfined particles of aggregate. In this type of test the observed deterioration of the aggregate is caused by the appropriate agent—freezing. However, such tests incorporate unrealistic conditions when the aggregates are presoaked in water or subjected to a "vacuum saturation" procedure prior to freezing. Such efforts to have the aggregate at a high state of saturation prior to test do not take account of the time period required for the aggregate to become critically saturated when in field concrete. In addition, such unconfined tests do not directly evaluate the type of concrete distress caused by expulsion of water into the mortar surrounding the aggregate. Although the test may appear to incorporate some aspect of saturability of the aggregate, the primary disruptive action involves only critical size effects since the aggregate is unconfined. The results obtained by such tests depend upon aggregate particle size, permeability, degree of saturation and porosity and rate of freezing and can be altered by the presence of soluble materials such as salts or alcohol. Whether an aggregate does or does not fail in tests of this type depends upon several arbitrarily selected test conditions and such tests may yield results of little importance to actual concrete performance.

Laboratory tests using concrete specimens containing the actual aggregates, cement, and mix design under consideration are by far the most satisfactory. However, such tests must be conducted in a manner consistent with the principles here discussed. To test concrete proposed for above grade structures or for pavements placed on free draining subbases and exposed to seasonal drying, a suitable procedure would include a period of air drying prior to wetting and subsequent freezing and thawing. Test methods using concretes containing presaturated aggregates without provision for preliminary drying can produce rapid and misleading deterioration in concretes not comparable with the actual service results.

Various examples of the inordinately severe conditions that have been incorporated in laboratory tests can be cited. In a recent series of cooperative freezing-and-thawing tests conducted by the Highway Research Board (**14**), the aggregates were presoaked for 7 days prior to use and the concretes were moist cured for 14 days without a period of air drying prior to freezing. This procedure was useful for the purpose of the test program, namely, to improve reproducibility but was unrealistically severe. A particular aggregate, selected because it was "poor," deteriorated rapidly during freezing-and-thawing tests even in air-entrained concrete, showing a 40 per cent loss in dynamic modulus of elasticity in about 20 to 35 rapid cycles of freezing and thawing in water. In contrast, the actual field performance of concretes containing this aggregate has been excellent in above grade structures (grain bins apparently showing no deterioration in 30 years) and at least marginal in pavements. Non-air-entrained pavements, particularly those laid directly on plastic subgrade soil, "began to show signs that the concrete was not durable when they had attained an age of 10 to 15 years" (**15**). The frost resistance of a concrete pavement containing this aggregate would undoubtedly be improved by air entrainment, a free-draining subbase, a smaller maximum size of aggregate, and the use of lower water-cement ratio concrete. These conditions, which are easily attainable, would be expected to improve the frost resistance of the concrete probably several fold. Satisfactory performance of such concrete as regards frost resistance might then be realized.

Laboratory freezing-and-thawing tests of concretes made with a particular chert aggregate also have demonstrated that results obtained with presaturated aggregate do not reflect actual performance in properly designed pavements. Non-air-entrained concretes made with saturated chert gravel and cured continuously moist failed rapidly on freezing and showed a 75 per cent loss in dynamic modulus in only 5 cycles of freezing and thawing. Air-entrained concretes also failed rapidly, showing a 70 per cent loss in dynamic modulus in 5 cycles. The ineffectiveness of air entrainment under such circumstances would be expected as the saturated chert aggregates fail because of the high internal hydraulic pressures and critical size effects previously discussed.

Testing of this aggregate in a more appropriate way, that is, air drying of the chert prior to use and a period of drying of the concrete prior to the freezing-and-thawing test, improved the performance of the concrete by about 80 fold. This chert has an excellent field service record in pavements placed on sandy soil or on a granular base where partial drying of the concrete occurs but has a poor service record in concretes placed on poorly draining heavy clay soils.

CONCLUSIONS

Based on the preceding discussions it is believed that the following major conclusions can properly be made:

1. The influence of aggregates on the durability of concrete depends upon the physical characteristics of the aggregates and certain properties of the mortar component of the concrete in a complex but understandable manner.

2. Certain test methods commonly used to evaluate the "durability" of aggregates for use in concrete are frequently inappropriate and misleading as regards actual field performance of the concrete.

3. Through proper design, based on the principles discussed, much can be done to improve significantly the actual performance of field concrete made with many aggregates rejected by commonly used tests.

REFERENCES

(1) H. S. Sweet, "Research on Concrete Durability as Affected by Coarse Aggregate," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 988 (1948).

(2) R. F. Blanks, "Modern Concepts Applied to Concrete Aggregate," *Proceedings*, Am. Soc. Civil Engrs., Vol. 75, p. 441, April (1949).

(3) T. C. Powers, "The Air Requirement of Frost-Resistant Concrete," *Proceedings*, Highway Research Board, Vol. 29, p. 184 (1949).

(4) R. L. Blaine, C. M. Hunt, and L. A. Tomes, "The Sorption Characteristics of Concrete Aggregates," *Proceedings*, Highway Research Board, Vol. 32, p. 298 (1953).

(5) D. W. Lewis, W. L. Dolch, and K. B. Woods, "Porosity Determinations and the Significance of Pore Characteristics of Aggregates," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 949 (1953).

(6) T. C. Powers, "Resistance to Weathering—Freezing and Thawing," ASTM *Special Technical Publication No. 169*, p. 182, 1956, Am. Soc. Testing Mats.

(7) G. Verbeck and P. Klieger, "Studies of 'Salt' Scaling of Concrete," Highway Research Board, *Bulletin 150*, p. 1 (1957).

(8) W. L. Dolch, "Studies of Limestone Aggregates by Fluid Flow Methods," *Proceedings*, Am. Soc. Testing Mats, Vol. 59, p. 1204 (1959).

(9) R. Rhoades and R. Mielenz, "Petrography of Concrete Aggregate," *Journal*, Am. Concrete Inst., p. 581, June, 1946, *Proceedings*, Vol. 42.

(10) A. E. Scheidegger, "The Physics of Flow Through Porous Media," p. 150, University of Toronto Press (1957).

(11) T. C. Powers, L. E. Copeland, J. C. Hayes, and H. M. Mann, "Permeability of Portland Cement Paste," *Journal*, Am. Concrete Inst., p. 285, Nov. 1954, *Proceedings*, Vol. 51.

(12) T. C. Powers, "Basic Considerations Pertaining to Freezing and Thawing Tests," *Proceedings*, Am. Soc. Testing Mats, Vol. 55, p. 1132 (1955).

(13) T. C. Powers and T. L. Brownyard, "Physical Properties of Hardened Portland Cement Paste," *Journal*, Am. Concrete Inst., p. 933, April (1947), *Proceedings*, Vol. 43.

(14) "Report on Cooperative Freezing and Thawing Tests of Concrete," Highway Research Board, *Special Report No. 47* (1959).

(15) Bert Myers, Iowa State Highway Commission, Private Communication, April 24, 1956.

THE EFFECTS OF EARLY FREEZING ON LOW-DENSITY AGGREGATE-TYPE CONCRETE*

By I. A. Benjamin[1] and G. D. Ratliff, Jr.[1]

Synopsis

A test program was initiated to study the effects of early freezing on low-density aggregate-type concrete. Specimens 3 in. thick and up to 10 sq ft in area, were frozen from 2 to 50 hr after placing. Expanded micaceous and siliceous aggregates were used. Temperatures in the slabs were recorded from the time they were placed until they began freezing. The actual age of the concrete was adjusted by a time-temperature function to obtain the equivalent 40 F constant-temperature curing age. Freezing damage was measured by two tests developed especially for this purpose: an interior strength and a surface strength test.

The data indicate that freezing after 10 hr has little effect on the interior strength. However, surface strength may be reduced to zero by early freezing, especially freezing before age 20 hr. Simulated exposure in a freezer did not reproduce the effect of outdoor exposure. The concrete which did not suffer damage due to early freezing was not damaged by exposure to the freezing-and-thawing weather. Micaceous and siliceous concretes showed comparable freezing damage.

Aggregate-type low-density concrete (oven-dry weight under 40 lb per cu ft) is made from micaceous and siliceous aggregates which have been expanded to loose-aggregate densities of 7 to 10 lb per cu ft. The gradation of the aggregate is governed by ASTM specification C 332 – 56 T.[2] The concrete is primarily used for roofs and its function differs from structural concrete, since it serves primarily as an insulating fill. The strength requirements are low: sufficient surface strength to adhere to the roofing, sustain foot traffic, and in some cases, carry a load on a short span. The concrete contains 30 to 40 per cent air, in the form of air bubbles in the matrix and air in the expanded aggregate. This compares with about 6 per cent air in normal air-entrained concrete.

There are no published reports on the performance of low-density concrete subject to early freezing. Since its strength requirements are not critical and since the high air content places it outside the range of experience of structural concrete, an investigation was started to determine if low-density concrete requires the same degree of cold-weather protection that regular concrete does. This report reviews the tests developed and some of the preliminary findings of the investigation.

A major problem in evaluating the

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Granco Steel Products, Subsidiary of Granite City Steel Co., St. Louis, Mo.

[2] Tentative Specification for Lightweight Aggregates for Insulating Concrete (C 332 – 56 T), 1958 Book of ASTM Standards, Part 4, p. 471.

freezing tolerance of any type of concrete is that of measuring the damage. Because of the special functional requirements of insulating fills: that they be strong enough to adhere to roofing materials and secure them against uplift, the surface strength is important. Therefore, a test was developed specifically to measure the loss of surface strength.

A test was also desired to measure the interior strength independent of the surface. For this test, a Proctor penetration instrument, designed for soil testing, was calibrated against standard 2 by 2-in. compression cubes. With the calibrated penetration test, it was possible to make in-place strength measurements over a prolonged period without destroying the slabs.

TABLE I.—MIX DATA.

	Expanded Aggregate	
	Micaceous	Siliceous
Mix, by volume of cement to aggregate.	1:6	1:6
Water, gal per sack cement.	19	12
Type cement.	III	III
Air entraining admixture.	powdered vinsol resin	liquid vinsol resin
Mean yield, per cent.	96	94
Mean wet density, lb per cu ft.	51	42

TEST PROCEDURE

The mix proportions used for all specimens are shown in Table I. The specimens were cast as simulated roof deck slabs, in forms made with galvanized corrugated steel bottoms and wooden sides $1\frac{5}{8}$ in. thick. The exposed surface area of the slabs varied from 1 to 10 sq ft. The average thickness was 3 in. Control slabs were made for each pour. The controls were air cured in the laboratory at a temperature of 60 to 70 F and relative humidity of 30 to 50 per cent.

Both simulated and actual outdoor exposures were used to investigate a wide range of concrete ages prior to freezing. Eleven slabs were poured in the laboratory and then taken outside at predetermined intervals. Eight slabs were poured outdoors, with the mix ingredients and the mixer at equilibrium with the outdoor air. All the outdoor slabs were placed at an elevation of 12 in. above the roof of the laboratory to permit cold air to circulate underneath them. They were left outside for the duration of a winter.

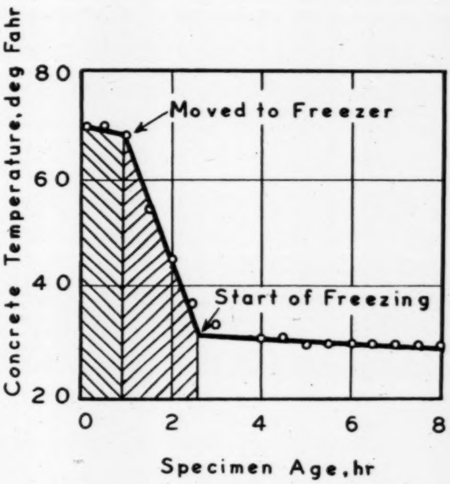


FIG. 1.—Typical Time-Temperature Curve for a Freezer Specimen, Showing Trapezoidal Areas Used to Adjust Actual Age Before Freezing to 40 F Equivalent Constant-Temperature Age.

A freezer capable of reducing the surface temperature of the concrete at the same rate that the outside air reduced it, was used for simulated exposures. Simulated and actual cold-weather exposures were duplicated by manually controlling the freezer to obtain equal time-temperature curves. After remaining in the freezer one night, the simulated exposure slabs were transferred to the roof alongside the outdoor exposure specimens.

The temperature of the freshly mixed concrete was measured with a bulb-type thermometer. After the concrete was placed in the forms, a thermocouple was inserted $\frac{1}{4}$ in. below the surface of the slab. The temperature was recorded at 15 to 30 min intervals, from the time the concrete was placed until the surface froze. The start of freezing is indicated on the time-temperature curve, Fig. 1, where the lower change of slope is due to the heat of crystallization of water.

EQUIVALENT CURING AGE

To take into account variations in curing temperature, the actual age of all the specimens was adjusted to an equivalent constant-temperature age. For this work a constant curing temperature of 40 F was chosen as the reference temperature. For example, concrete curing for 10 equivalent hours presumably has the same strength properties as concrete cured for 10 hr at 40 F. This temperature was chosen since it is a commonly specified minimum ambient air temperature at which insulating concrete should be placed during the winter without providing special protection.

The mathematical function suggested by Saul (**1**)[3] was used to derive the equivalent 40 F age. This function, which has been successfully used to reflect experience with British and Finnish cements was assumed to be generally applicable to American cement. The function is:

$$N = 0.556\,(t - 14)a \quad \text{........}(1)$$

where:

N = time-temperature function of strength, and
a = curing time at temperature t.

The general equation, Eq 2, was derived by integrating a trapezoidal area with the Saul function for the ordinate. This equation makes it possible to divide the time-temperature curve into trapezoidal elements and determine the equivalent age of specimens prior to freezing:

$$A_c = 1.92 \times 10^{-2}\,(t_1 + t_2 - 28)A \quad \text{.........}(2)$$

when t_1 and $t_2 > 32$ F
where:

A_c = equivalent curing time at 40 F,
A = actual curing time,
t_1 = initial temperature, and
t_2 = final temperature.

In Fig. 1, a typical time-temperature curve is broken into trapezoidal elements to illustrate the method. The equivalent 40 F curing age prior to freezing, calculated for the hatched areas, is 4.2 hr.

To measure the equivalent curing time while the concrete was frozen, the formula was modified to:

$$A_c = 3.85 \times 10^{-3}\,(t_1 + t_2 - 10)A \quad \text{.........}(3)$$

when t_1 and $t_2 < 32$ F.

This formula, based on the Saul function modified as suggested by Nykanen and Pihlajavaara (**2**), reflects the fact that concrete has some limited strength gain even while frozen. Powers (**3**) has indicated that evaporable unfrozen water may exist in concrete down to −108 F.

METHODS OF MEASURING FREEZING DAMAGE

Several preliminary studies were devoted to finding ways of measuring the different types of freezing damage. It is known from field experience that the surface and interior damage are of a different nature. Therefore, compression tests, measures of dilation, or loss of sound velocity were not considered satisfactory since they show the average change in strength, but not necessarily the loss of surface or interior strength.

A nondestructive indentation test, previously reported by Benjamin and Ratliff (**4**), was used to measure the interior strength of a slab at successive intervals. Initial exposure effects could be observed on the long-time strength gain of the interior of the slabs.

---

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

When it became apparent that early freezing reduced the ability of the concrete to hold down a roof, a test was adopted to measure the surface strength by duplicating qualitatively an actual roofing operation. A small puddle of hot bitumen was poured on the concrete slab. A wooden block was immediately embedded in the liquid bituminous material before it hardened. The block was $2\frac{1}{4}$ by $2\frac{1}{4}$ in. by $1\frac{5}{8}$ in. thick, having a contact area of 0.035 sq ft. A hook was screwed into the block for applying a load. After the bitumen hardened enough to insure good bond to the wooden block, 30 min, the excess bitumen was trimmed away. The block was then pulled from the concrete by lifting it vertically by means of a spring scale (Fig. 2). The lifting was carried out within a 10-sec period. Generally the blocks separated as a result of a tensile failure in the top $\frac{1}{8}$ in. of the concrete. The test was not considered satisfactory when more than 10 per cent of the contact surface showed asphalt failure.

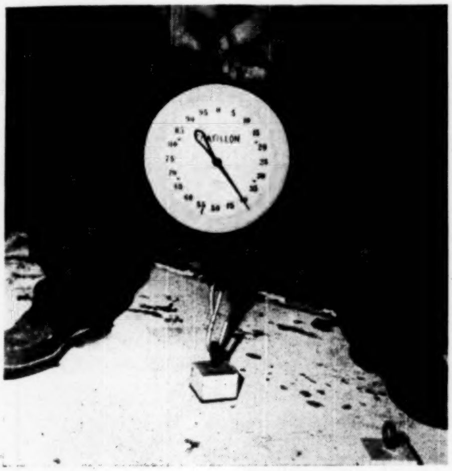


FIG. 2.—Method of Measuring Surface Strength.

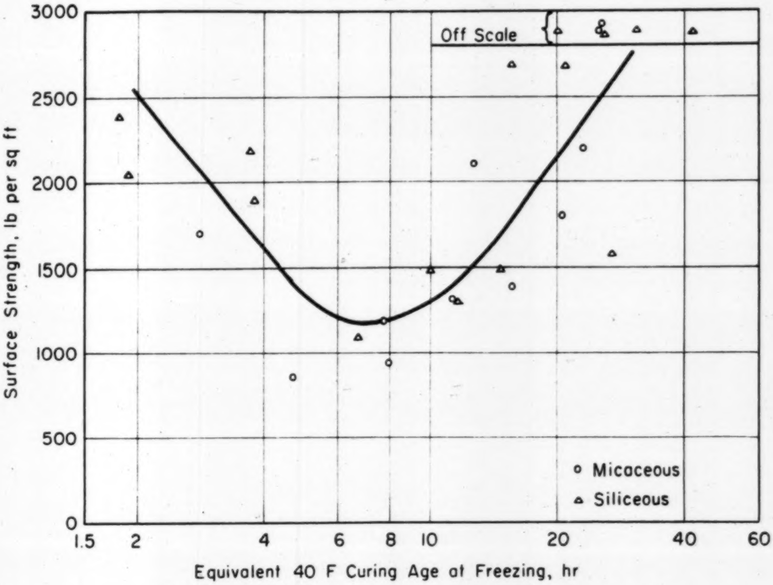


FIG. 3.—Effect of Simulated Exposure on Surface Strength.

Using this test, control specimens of mature concrete failed at a load of 200 lb on the spring scale, equal to a surface strength of 5500 lb per sq ft. In testing damaged surfaces, a 100-lb scale was used to obtain greater accuracy in the low ranges. A series of eight different concrete mixes, having a range of surface strength from 670 to 5820 lb per sq ft, was tested to evaluate the reproducibility of the surface strength test. A pair of specimens was tested from each mix of concrete. The maximum variation from the average strength for each pair of tests was 9.8 per cent. The average variation was 5.8 per cent.

TEST RESULTS

Surface Damage:

Surface damage on low-density concrete is easily observed and felt. In some instances, a hard crust of concrete about $\frac{1}{8}$ in. thick scales off of the top. In other instances, the concrete does not scale, but becomes soft and powdery and can be readily indented with the thumb. It has the appearance of loose aggregate without cement.

Surface damage seemed to be a function of at least two variables: type of exposure and equivalent age at freezing. Figures 3 and 4 show the relationship between the equivalent age at freezing and surface strength for both simulated and actual exposures. Although both methods of freezing lowered the surface strength, actual outdoor exposure lowered it more than simulated exposures. The minimum surface strength of the freezer specimens was 800 lb per sq ft, whereas three of the specimens placed outdoors had a surface strength as low as 100 lb per sq ft. Simulated freezing seemed to be more damaging at equivalent age 6 hr, than at any other age. Specimens frozen outside did not show a similar dip in the freezing-age versus strength curve but a well defined increase in resistance to freezing as the equivalent age at freezing is increased.

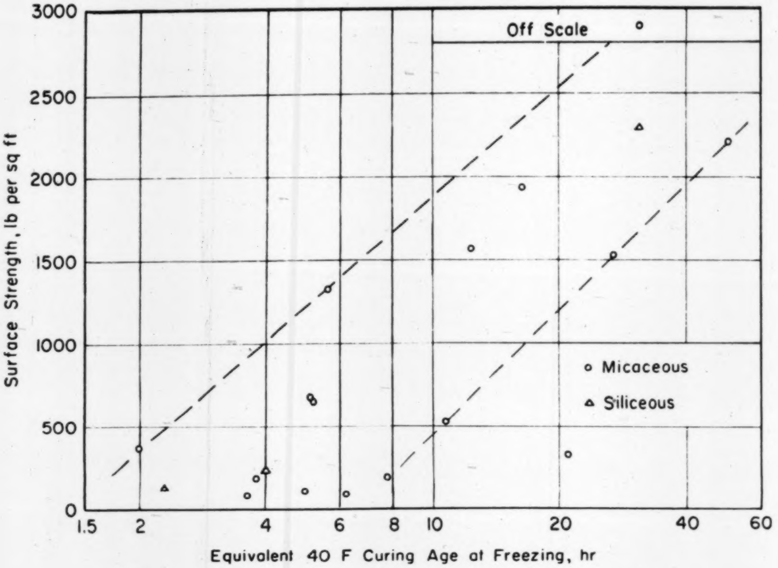


FIG. 4.—Effect of Outdoor Exposure on Surface Strength.

There is considerable scatter in the data on Fig. 4, the outdoor exposures. Part of the scatter is the result of variations in factors such as solar radiation, wind, humidity, and precipitation.

Specimens that were not damaged on the surface by the initial freezing remained undamaged for the duration of the winter.

Interior Strength:

Figure 5 shows the interior strength at 28 days equivalent curing for both types of low-density concrete. Figure 6 shows the strength of the micaceous aggregate concrete as a percentage of the control strength at 100 days equivalent curing. Siliceous aggregate concrete data were not available for this comparison. Both graphs show that interior strength is not appreciably reduced by freezing after it attains an equivalent curing age of 10 hr. This would indicate that the protection required to prevent surface damage is more than enough to prevent interior damage.

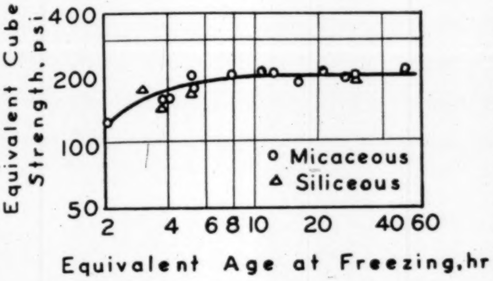


FIG. 5.—Effect of Outdoor Exposure on Cube Strength Obtained by Converting Equivalent 28-day Proctor Penetration Resistance Readings.

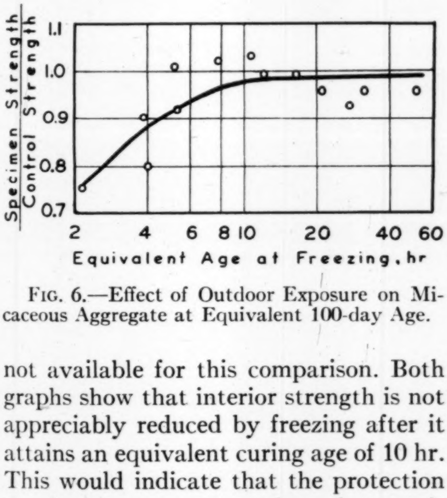


FIG. 6.—Effect of Outdoor Exposure on Micaceous Aggregate at Equivalent 100-day Age.

DISCUSSION

Regular concrete does not appear to be as vulnerable to surface damage as low-density concrete. The 98 per cent loss of surface strength of low-density concrete subjected to early freezing is easily apparent. Such severe damage should be apparent with regular-density concrete too; however, it has not been reported.

Nykanen and Pihlajavaara (2) obtained a 20 per cent loss of strength for structural concrete specimens frozen after curing at any age from 2 to 40 equivalent hours at 40 F. As shown in Figures 5 and 6, low-density concrete is not damaged after curing for 10 hr at 40 F. The difference in the freezing resistance of insulating concrete, from that of structural concrete, may be due to the difference in density of the concrete, 30 versus 140 lb per cu ft or to the difference in the aggregate strength and its relative contribution to the strength of the concrete.

Tests by Klieger (5) have shown that concrete cured at 40 F will be stronger than that cured at 70 F. Since the interior strength, in Fig. 6, was compared with controls cured indoors, the effect of outdoor winter curing may tend to compensate for freezing damage.

Surface moisture may be an important variable. The vapor pressure buildup, inherent when wet specimens are put in a freezer, may account for the difference in surface damage between simulated and actual exposures. As further evidence, it was observed that the underside of the specimens was not damaged by freezing, even when the top surface was severely damaged. The bottom surface was protected by a galvanized steel form which prevented moisture loss.

Moisture in any form seems to be important. For example, field experience indicates that ponding of water on exposed low-density roofs will lead to deterioration due to cycles of freezing and thawing. On the other hand, the well-drained specimens in this investigation remained sound after 17 cycles of freezing and thawing during the winter they were exposed.

CONCLUSIONS

If low-density concrete freezes at an equivalent age of less than 20 hr, damage will occur on the top surface. Scaling or crumbling of the surface concrete may result. The cohesive strength of this loose material may be too low to hold down roofing.

The interior strength is not appreciably affected unless the concrete freezes before an equivalent age of 10 hr.

Freezer exposures do not simulate actual outdoor exposures. There was no difference in these preliminary tests in the performance of micaceous and siliceous aggregates. Cycles of freezing and thawing did not damage specimens which were not damaged by the initial freezing.

Further work is needed on other types of low-density concrete, other mix proportions, and other brands and types of cement. The influence of vapor pressure on freezing resistance seems to also warrant further investigation.

REFERENCES

(1) A. G. A. Saul, "Principles Underlying the Steam Curing of Concrete at Atmospheric Pressure," *Magazine of Concrete Research*, Vol. 2, p. 6, section 127 (1951).

(2) A. Nykanen, and S. Pihlajavaara, "The Hardening of Concrete Under Winter Concrete Conditions," The State Institute for Technical Research, Helsinki (Finland) (1958).

(3) T. C. Powers, "Physical Properties of Hardened Portland Cement Paste, Part 8," *Journal*, Am. Concrete Inst., April, 1947.

(4) Irwin A. Benjamin and George D. Ratliff, Jr., "An In-Place Strength Test for Low-Density Concrete," ASTM BULLETIN, No. 241, Oct., 1959, pp. 23–26.

(5) Paul Klieger, "Effect of Mixing and Curing Temperature on Concrete Strength," *Journal*, Am. Concrete Inst., June, 1958.

EFFECT OF HIGH TEMPERATURES ON CONCRETES INCORPORATING DIFFERENT AGGREGATES* †

By N. G. Zoldners[1]

Synopsis

Changes in physical properties of concrete beams and cylinders made with gravel, sandstone, limestone, and expanded slag aggregates were studied after exposure to various temperatures ranging from 100 to 800 C.

Results indicated that the residual compressive strength of concrete increased up to 10 per cent when the specimens were heated up to 300 C, whereas the flexural strength decreased 34 per cent with limestone and 55 per cent with gravel aggregate. After 500 C exposure, losses in strength of corresponding concretes were 19 to 24 per cent in compression and 58 to 80 per cent in flexure.

In temperatures over 500 C, the residual strength of all types of concrete decreased sharply due to dehydration of chemical constituents in the cement paste.

Results from the four types of concrete investigated indicate that limestone concrete gave the best performance up to 700 C, when decomposition of carbonates on the surface of test specimens was observed.

Concrete is recognized as an excellent fire-resistive building material.

Building codes specify the fire protection requirements for specific structural members in terms of fire resistance ratings based upon tests made in accordance with ASTM standards. Different structural assemblies have been fire-tested and given hourly ratings in a fire-retardant classification (**1**).[2]

Over the years, accumulated experience obtained from fire-endurance and heat-transmission tests on structural assemblies has provided sufficient data for fire protection, but little research has been done on the behavior of concrete subjected to high temperatures.

With the present-day uses of concrete in certain building construction and industrial installations, in aircraft engine test cells, and in turbo-jet runways, atomic reactors, and missile launching pads, additional information is required on the effect of high temperatures.

Basic research on physical properties under varying thermal conditions could provide this information.

Fundamental research studies of the

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

† Published by permission of the Director, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

[1] Head, Construction Materials Section, Mineral Processing Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

fire resistance of concrete have been started recently at the new Fire Research Laboratories of the Portland Cement Assn. (**2**,**3**).

An investigation on concrete properties in the lower temperature ranges has been conducted at the University of Wisconsin (**4**).

Research work in this field was begun at the Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada, early in 1958.

A list of references and related publications is included at the end of this paper.

TABLE I.—CHEMICAL ANALYSIS AND COMPOUND COMPOSITION OF CEMENT.[a]

Chemical Analysis, per cent		Compound Composition, per cent	
SiO_2	21.05		
Al_2O_3	5.66	C_3S	47.1
Fe_2O_3	3.03	C_2S	24.9
CaO, combined	62.22	C_3A	9.9
CaO, free	0.83	C_4AF	9.2
MgO	2.75	$CaSO_4$	4.2
SO_3	2.47	MgO Ignition loss Alkalies	4.6
Na_2O	0.48		
K_2O	0.56		
Ignition loss	0.81		
Total	99.86	Total	99.9

[a] Chemical analysis by Canada Cement Company Limited, Hull, Quebec, Canada.

SCOPE OF RESEARCH

Primarily, this project was undertaken to study the effect of high temperatures on concrete prepared with different aggregates. No attempt was made to evaluate the performance of concrete structural members in fire exposures.

Evaluation of fired concrete specimens by determining the compressive strength only is misleading. Multiple methods of evaluating the physical conditions of concrete were applied in this project.

Flexural and compressive strengths of beams and cylinders along with changes of weight, volume and ultrasonic pulse velocities were studied and ruptured concrete surfaces were microscopically examined, to determine the degree of concrete deterioration after exposure at elevated temperatures between 100 and 800 C.

Thermal conductivity values and specific heat data on each of the four types of concrete were used to compare the thermal properties of the materials.

It is hoped that information obtained in this investigation may contribute to the knowledge of concrete and its fire-resistance properties.

MATERIALS

Cement:

A type 1 portland cement of Canadian manufacture was used in this project. The chemical analysis and compound composition of this cement is shown in Table I.

Aggregates:

Identically graded fine and coarse aggregates were prepared from the same materials—gravel, sandstone, limestone, and expanded slag.

Gravel was from an operating pit of a glacial-fluvial deposit at Shawville, Quebec. This gravel, a heterogeneous mixture, is being used in ready-mixed concrete in Ottawa, Ont.

Sandstone was from a quarry in Potsdam formation at Beauharnois, Que. The rock is clean, white, and well indurated.

Limestone was obtained from an Ottawa quarry. The rock is a dark, fine-grained high-calcium variety of limestone, typical of parts of the local Trenton formation.

Expanded slag, a commercial by-product of the steel industry, was obtained from Hamilton, Ont.

A detailed petrographic study was made on each of the four types of aggregate used in the concrete to be tested.

TABLE II.—PETROGRAPHY OF AGGREGATES USED IN CONCRETE.

Aggregate, per cent		Description
GRAVEL		
Metamorphic rock (gneiss, schist, minor calcite)	45	Heterogeneous aggregate of gneissic, schistose, granitic, and mafic igneous or metamorphic rock material with lesser amounts of sedimentary limestone and sandstone, typical of many glacial-fluvial deposits
Granitic and minor basic igneous rock	42	
Limestone and clayey limestone	12	
Sandstone	1	
SANDSTONE		
White to bluish	88	Relatively pure, white sandstone or orthoquartzite, consisting of firmly cemented fine, equant, rounded quartz grains. Twelve per cent is buff-weathering.
White, buff streaks	12	
LIMESTONE		
High-calcium, hard	80	Fairly pure high-calcium, fine-grained limestone containing a few fossils. Shaly portions contain dispersed clay.
Shaly (15 per cent clay)	20	
EXPANDED SLAG		
Gray-white	58	Two phases: (1) Gray-white, relatively dense, containing akermanite and monticellite.
Buff, porous	42	(2) Buff, porous, containing merwinite.

TABLE III.—GRADING OF AGGREGATES.

Coarse Aggregate			Fine Aggregate		
Sieve Size	Per cent Passing		Sieve Size	Per cent Passing	
	Standard	Expanded Slag		Standard	Expanded Slag
¾-in.	100.0	...	No. 4 mesh	100.0	100.0
½-in.	56.0	...	No. 8 mesh	90.0	80.0
⅜-in.	37.5	100.0	No. 16 mesh	67.5	55.5
No. 4 mesh	0.0	0.0	No. 30 mesh	42.5	35.0
			No. 50 mesh	20.0	20.0
			No. 100 mesh	6.0	10.0

When necessary, X-ray diffraction and chemical analyses were carried out. Table II summarizes the results of these studies.

Gravel, sandstone, and limestone aggregates were crushed and screened into size fractions, and then recombined according to the grading shown in Table III, under column marked "Standard."

Expanded slag coarse aggregate was used as received, whereas fine aggregate was recombined as shown in Table III.

Some physical properties of the four aggregates and proportions of coarse to fine aggregate in concrete mixes are shown in Table IV.

PREPARATION OF TEST SPECIMENS

Concrete Mixes:

All concrete mixes were prepared with the same cement factor—480 lb of cement per cu yd of concrete.

TABLE IV.—PHYSICAL PROPERTIES AND MIX PROPORTIONS.

	Gravel		Sandstone		Limestone		Expanded Slag	
	C.A.[a]	F.A.[a]	C.A.	F.A.	C.A.	F.A.	C.A.	F.A.
Specific gravity (bulk, SSD[b] base)	2.71	2.70	2.62	2.60	2.68	2.66	1.76	2.02
Absorption, per cent by weight	0.78	1.22	1.1	1.5	0.92	1.22	18.9	20.0
Mix proportions, per cent by weight	59	41	52	48	52	48	25	75

[a] C.A. = coarse aggregate.
F.A. = fine aggregate.
[b] SSD = saturated, surface-dry.

TABLE V.—CONCRETE MIX PROPORTIONS.

Type of Concrete Aggregate	Average Mix Proportions per 1 cu yd					Mix Characteristics			
	Cement, lb	SSD[a] Aggregate		Water, lb	Admixture	Water-Cement ratio	Slump, in.	Air, per cent	Unit Weight, lb per cu ft
		Fine, lb	Coarse, lb						
Gravel	480	1345 41 per cent	1935 59 per cent	300	nil	0.62	2½	1.5	150.5
Limestone	480	1550 48 per cent	1670 52 per cent	314	nil	0.65	2½	1.1	148.8
Sandstone	480	1450 48 per cent	1570 52 per cent	330	nil	0.69	2	2.9	141.0
Expanded slag	480	1357	452	475	Darex 6 oz	...	0	9.0	102.5

All but one of the mixes were nonair-entrained with a consistency of 2 to 2½ in. slump. In mixes with expanded slag aggregate, an air-entraining admixture was added for workability.

Mix proportions and other data are shown in Table V.

Test Specimens:

Ten 1.6-cu ft batches were prepared for each of the four types of concretes.

Normally, from each batch either 12 beams (3½ by 4 by 16 in.), 18 cylinders (4 by 8 in.), or 2 slabs (3 by 13 by 17½ in.) were molded.

From every batch two sets of fire-test specimens and one set of control specimens were selected. A set of fire-test specimens consisted of either four beams or six cylinders. A control set consisted of either four beams or three cylinders.

The first batch was used to prepare beam and cylinder specimens for preliminary firings and for standard test specimens.

From the last batch, two slabs were prepared for thermal conductivity, and 12 spare test cylinders were made from the remaining material.

Table VI shows a breakdown of all test specimens for one type of concrete, as prepared from each batch.

This schedule allowed eight test firings to be made at eight different temperatures, using a set of four beams or six test cylinders for each firing. A sufficient number of specimens was available to conduct two preliminary firings for each series in order to establish the proper heat control in the furnace.

On the 4-in. sides of the beams, two

TABLE VI.—SUMMARY OF TEST SPECIMENS.

For one test series only

Number of Mix Batches	Type of Test Specimens	Number per Batch	Allocation of Specimens					Total Number of Specimens
			Fire Tests	Control Specimens	Standard Specimens	Reference Specimens	Thermal Conductivity	
1	Beams	6	3	...	...	3	...	6
	Cylinders	12	...	...	12	...	...	12
4	Beams	12	8	4	...	...	...	48
4	Cylinders	18	12	3	...	3	...	72
1	Slabs	2	...	...	...	...	2	2
	Cylinders	12	12	...	...	...	...	12
Total number of specimens								152

TABLE VII.—PROPERTIES OF HARDENED CONCRETE.

Type of Concrete Aggregate	Standard Moist-cured Specimens					12 Month Room-dried Control Test Specimens					
	SSD[a] 7 days Density, lb per cu ft	Average Compressive Strength of 4 by 8 in. Test Cylinders				Room-dry 4 by 8 in. Cylinders		3½ by 4 by 16-in. Test Beams			
		7 days, psi	14 days. psi	28 days, psi	12 months, psi	Density, lb per cu ft	Compressive Strength, psi	Loss in Weight, per cent	Free Moisture Retained, per cent	Modulus of Rupture, psi	Beam End Compressive Strength, psi
Gravel	151.6	2340 67 per cent	3110 89 per cent	3475 100 per cent	4517 130 per cent	143.9	4008	5.1	0.5	763 14.6 per cent	5288 100 per cent
Limestone	150.0	2719 68 per cent	3465 87 per cent	4003 100 per cent	4810 120 per cent	142.8	4160	4.9	0.5	719 15.4 per cent	4667 100 per cent
Sandstone	144.4	1900 68 per cent	2315 83 per cent	2772 100 per cent	4080 147 per cent	136.9	3542	5.2	0.4	588 13.9 per cent	4247 100 per cent
Expanded slag	111.2	934 64 per cent	1175 80 per cent	1461 100 per cent	not tested	96.5	1597	13.2	0.8	397 19.9 per cent	1998 100 per cent

[a] Saturated, surface-dry.

stainless steel plugs were cast 10 in. apart to provide reference points for length measurements.

Curing Test Specimens:

The twelve test cylinders from the first batch were stored under standard moist-curing conditions for 7-, 14-, 28-day, and 12-month compression tests.

All other test specimens, after 7-day initial standard moist-curing, were kept in a dry-storage room for 1 yr until tested.

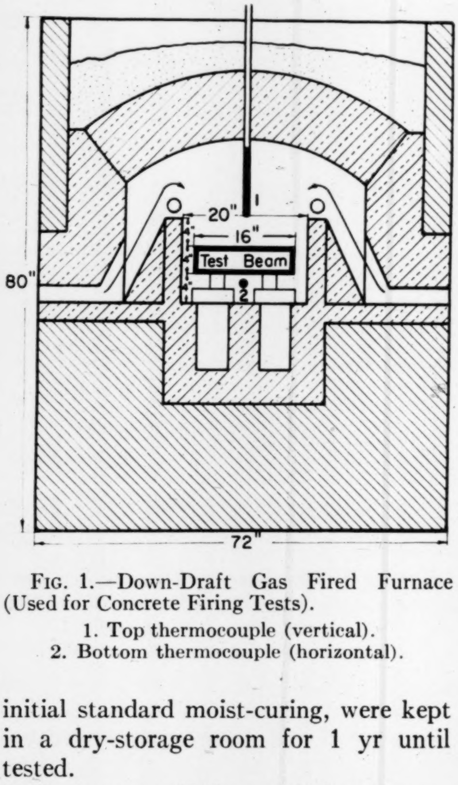


FIG. 1.—Down-Draft Gas Fired Furnace (Used for Concrete Firing Tests).

1. Top thermocouple (vertical).
2. Bottom thermocouple (horizontal).

Three test cylinders and three beams from the four series were selected as reference specimens for density and moisture-change determinations in the dry-storage room. These specimens were weighed regularly once a week, providing a moisture-loss record.

Properties of Hardened Concrete:

Density values at the beginning and at the end of the 12-month room-drying period, as well as the amount of moisture lost and remaining in the room-dried control test specimens, are shown in Table VII. Also shown are compressive strengths of standard moist-cured and of room-dried concrete test specimens. The values are the average of three results obtained for each set of beams or cylinders.

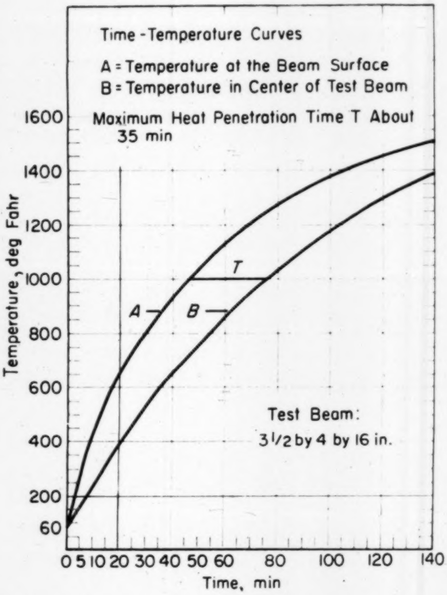


FIG. 2.—Heat Penetration—Preliminary Firing Data.

HIGH-TEMPERATURE EXPOSURE

Fire-Test Installations:

A gas-fired furnace of 10 cu ft capacity was used for the firing tests. Three exchangeable gas burners on each side of the furnace, coupled with air valves and draft regulators, provided temperature control in the heating chamber.

Two thermocouples, one in the middle of the chamber and the other between test specimens, were connected to an automatic temperature recorder to provide a continuous record of the furnace temperature.

A vertical section of the furnace is shown in Fig. 1. The dimensions of the furnace chamber were: width, 20 in.; depth, 42 in.; and height at the crown, 24 in.

Thermal Distribution:

To determine the heat distribution in the furnace and penetration within test specimens, a preliminary firing was conducted. Six chromel-alumel thermocouples were used with a potentiometer. Four of these units were installed to measure thermal differentials between the outside and the inside of a beam and of a cylinder, one was placed between test specimens, and the other indicated the temperature in the middle of the furnace.

Data obtained from the preliminary firing was sufficient to establish two time-temperature curves for a concrete test beam, one representing the temperature rise on the surface and the other the temperature rise in the center, recording the heat penetration, as shown in Fig. 2.

These curves indicate that the temperature rise within the test specimen lagged behind the furnace temperature rise by about ½ hr. When the furnace reached the predetermined temperature, 1 hr of heat-soaking period was specified for the test specimens to reach thermal equilibrium.

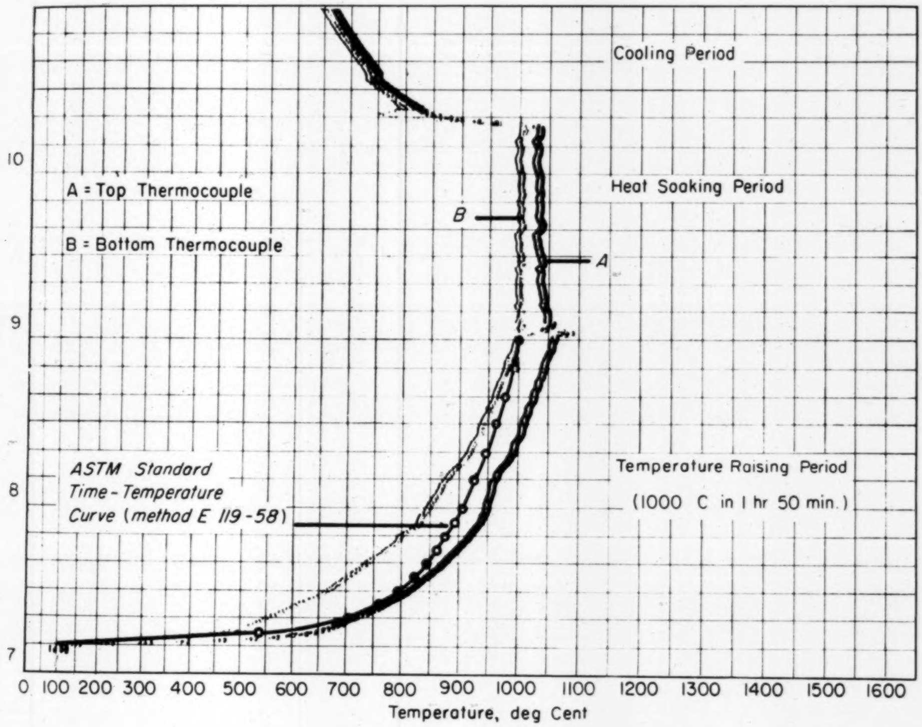


FIG. 3.—Preliminary Firing—Temperature Record.

In a trial run, with two thermocouples located as shown in Fig. 1, a temperature of 1000 C was recorded by the lower thermocouple in about 2-hr time. The recorded curves obtained indicated that the temperature rise in the furnace followed closely the standard-time temperature curve, as specified by ASTM

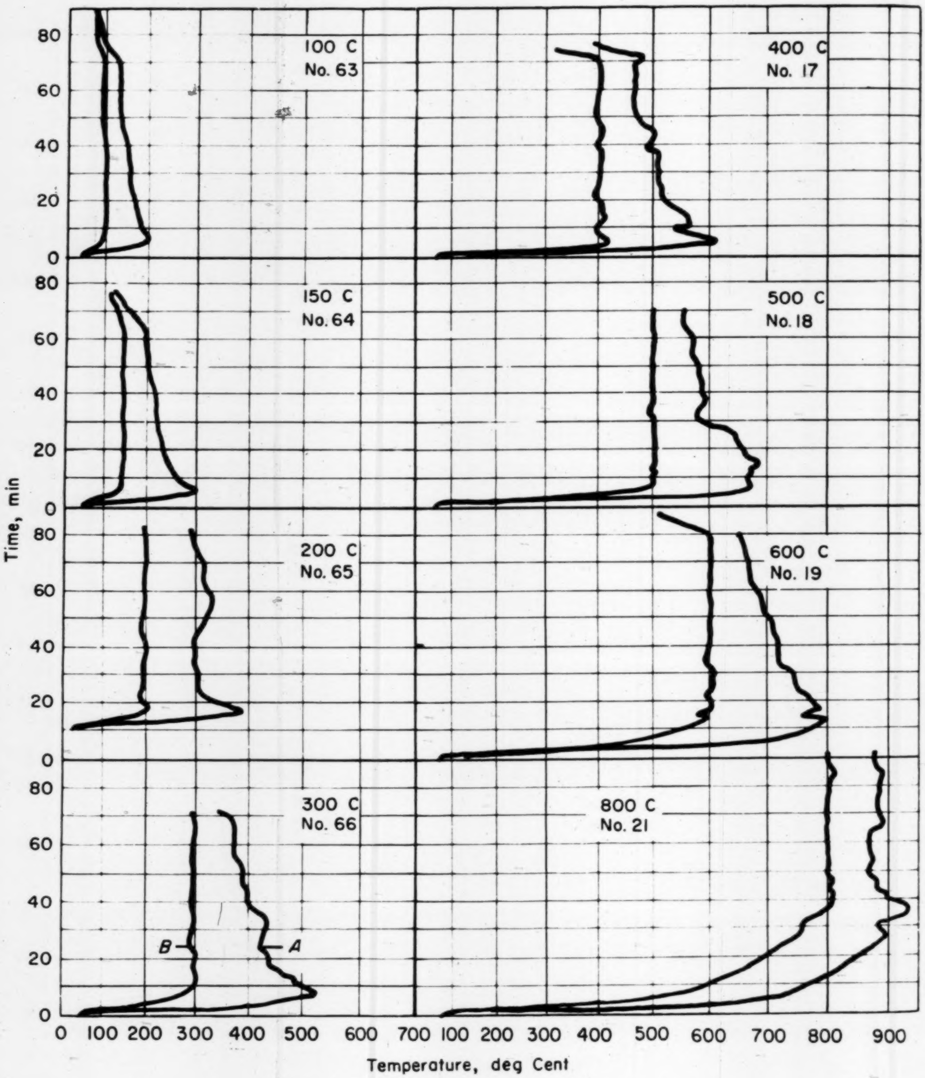


FIG. 4.—Furnace Temperature Records.
A. Top thermocouple.
B. Bottom thermocouple.

Standard Methods E 119 – 58[3] (see Fig. 3).

Fire-Test Procedure:

The heating chamber of the gas furnace provided space for either 12 cylinders (4 by 8 in.) or 4 beams (3½ by 4 by 16 in.) at one time.

Cylinders were placed in groups of three in four rows, with top ends protected from the direct flame by refractory brick. Six cylinders from each of two concrete test series were fired at the same time.

---

[3] Standard Methods of Fire Tests of Building Construction and Materials (E 119 – 58), 1958 Book of ASTM Standards, Part 5, p. 969; Tentative Revision of Methods E 119, 1959 Supplement to Book of ASTM Standards, Part 5, p. 201.

Beams were placed horizontally on their $3\frac{1}{2}$-in. sides (see Fig. 1), with the steel reference plugs located on both 4-in. sides. All four beams were from the same concrete batch.

Fire test specimens were exposed from room temperature to 100, 150, 200 C and then by 100 C increments for each firing up to 800 C. After reaching the predetermined end-temperature, specimens were heat-soaked for 1 hr.

Some typical temperature records for different firing ranges, provided by the two thermocouples, are shown in Fig. 4.

The lowest temperature was given by the bottom thermocouple, which recorded temperatures that were from 100 to 300 C lower than the upper one. The bottom temperature was the nominal temperature of the test environment and was held constant at the predetermined value. The upper temperature decreased gradually, approaching equilibrium at the end of the heat-soaking period.

Immediately after firing, three of the six test cylinders of each series were removed from the furnace, quenched in water for 5 min, and were then transferred to a drying oven at 50 C for 18 hr. The remaining cylinders were left overnight in the closed furnace to cool slowly. The next morning all fired specimens were placed in a desiccator to bring them to room temperature in 4 hr until tested.

The beam specimens were not quenched.

PHYSICAL TEST PROCEDURES AND METHODS

Physical Tests:

To evaluate the effect of high temperatures on concrete, the following investigations were carried out:

1. Weight determinations,
2. Length measurements,
3. Pulse velocity determinations,
4. Flexural and compressive tests on beams,
5. Compression tests on cylinders,
6. Thermal properties of concrete,
7. Petrographic examinations of the heated and crushed test specimens.

Fire test specimens were weighed, and the length and pulse velocity of beams were measured, before and after heat exposure.

All fire test specimens were preconditioned by air-drying for a period of 12 months.

Methods:

Test specimens were weighed with a probable accuracy of ±0.001 lb (0.5 g).

Length measurements were made on beam specimens only. Reference points were drilled in the embedded stainless steel plugs 10 in. apart. A Whittemore strain gage was used, having a dial reading of 0.0001 in.

Ultrasonic pulse velocity was determined by a type UCT electronic concrete tester.[4] This instrument operates at a 100 kc per sec frequency with 0.1 μsec sensitivity.

Concrete flexural strength was tested on the $3\frac{1}{2}$ by 4 by 16-in. beams according to ASTM Standard Method C 78 – 57,[5] using simple beam, third-point loading. Specimens were tested in a Tinius Olsen Super "L" compression testing machine, the sensitivity of which in the 1200-lb range was 2 lb per dial division.

Portions of beams broken in flexure were tested for compression according to

---

[4] Manufactured by A. E. Cawkell, Electronic Engineers (6–7, Victory Arcade, The Broadway, Southall, Middx., England).

[5] Standard Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 – 59), 1959 Supplement to Book of ASTM Standards, Part 4, p. 667.

TABLE VIII.—TEST RESULTS ON CONCRETE AFTER EXPOSURE TO HIGH TEMPERATURES.

No ultrasonic pulse velocity determinations were made on expanded slag concrete.

Test Temperature, deg Cent	Fired Strength in per cent of Original Average Strength															
	Ultrasonic Pulse Velocity				Beam Flexural Strength				Beam Ends Compressive Strength				Cylinder Compressive Strength			
	Gravel	Sandstone	Limestone	Expanded Slag	Gravel	Sandstone	Limestone	Expanded Slag	Gravel	Sandstone	Limestone	Expanded Slag	Gravel	Sandstone	Limestone	Expanded Slag
20	100	100	100	...	100	100	100	100	100	100	100	100	100	100	100	100
100	99	99	...	...	83	93	...	...	93	99	...	...	100	...	...	...
150	96	98	98	...	68	85	85	...	92	91	95	...	97	102	95	...
200	88	94	96	...	63	75	73	68	97	96	91	99	99	112	98	105
300	74	82	92	...	45	55	66	34	102	107	110	84	103	97	94	77
400	69	65	85	...	26	35	62	16	82	80	91	64	85	84	94	73
500	47	54	69	...	20	26	42	18	71	75	89	65	76	71	81	67
600	38	29	54	...	19	12	29	16	67	49	66	68	46	43	52	71
700	30	24	46	...	12	8	18	14	33	35	46	62	26	30	42	49
800	...	...	...	...	...	...	...	14	...	...	...	28	15	21	24	30

TABLE IX.—LENGTH AND WEIGHT CHANGES OF CONCRETE AFTER FIRE EXPOSURE.

Exposure Temperature, deg Cent	Weight Loss of Room-Dry Concrete, per cent				Permanent Length Changes of Concrete Beams After Exposure, 10^{-3} in. per in.			
	Gravel	Sand	Limestone	Expanded Slag	Gravel	Sand	Limestone	Expanded Slag
100	0.04	0.06	0.05	...	−0.01	−0.02	...	...
150	0.30	0.30	0.30	...	−0.01	−0.03	...	...
200	1.17	1.14	1.16	1.80	−0.03	−0.10	−0.05	0.19
300	1.86	1.70	1.80	3.07	+0.02	−0.11	−0.13	−0.32
400	2.14	1.90	1.97	4.07	+0.5	+0.2	+0.3	−0.27
500	2.60	2.19	2.48	4.50	+2.2	+1.0	+1.1	−0.40
600	3.42	2.94	3.20	4.95	+4.2	+5.0	+3.1	−0.34
700	4.61	3.23	4.6	5.77	+6.0	+8.8	+5.1	−0.26

TABLE X.—THERMAL PROPERTIES OF CONCRETE.

Type of Concrete Aggregate	Thermal Conductivity			Room-dry Concrete Density, lb per cu ft	Mean Specific Heat 25 to 400 C, Btu per lb per deg Fahr	Heat Diffusivity, sq ft per hr
	Mean Temperature		Coefficient *K*, Btu in. per hr sq ft deg Fahr			
	deg Cent	deg Fahr				
Gravel	100	212	10.60	...	...	0.032
	164	327	10.60	143.9	0.229	...
	402	756	8.95	...	...	0.027
Limestone	164	327	6.75	142.8	0.236	0.020
	394	740	8.04	...	...	0.024
Sandstone	95	202	15.80	...	...	0.050
	169	336	15.75	136.9	0.233	...
	406	763	10.64	...	...	0.033
Expanded slag	182	359	3.51	96.5	0.222	0.016
	398	749	3.45	...	...	...

TABLE XI.—PHYSICAL FEATURES OF HEAT-EXPOSED CRUSHED CONCRETE CYLINDERS AND BEAMS.

Aggregate	Temperature, deg Cent	Aggregate-Mortar Bond	Ruptured Surface	Conclusions
Gravel	200 to 400	Strong; few boundary cracks.	2 to 5 per cent across small[a] aggregate particles, least at 400 C.	Start of bond yield at 400 C; some aggregate may be expanding.
	500, 600	Weakened; yielding along aggregate surfaces; fine mortar cracks.	1 to 2 per cent across particles less than $\frac{3}{8}$ in.; quench 600 C; outer rim separating.	Cement decomposes; some aggregate particles expanding.
	700, 800 (See Figs. 5 and 6)	Poor; aggregate particles loose, concrete exfoliating.	1 to 2 per cent across friable particles; powdery cement; micaceous particle pop-outs.	Mortar bond destroyed by heat; pop-outs from expanding particles.
Limestone	200 to 400	Strong; few boundary cracks.	5 to 10 per cent across small[a] aggregate.	Heating: no visible effect.
	500, 600	Weakened; yielding along aggregate surfaces; fine mortar cracks.	5 per cent across small[a] aggregate at 500 C; less than 1% at 600 C quenched.	Heating decomposes cement; quench: fine cracks at 600 C.
	700, 800 (See Figs. 7 and 8)	Weak; abundant cracks; powdery cement; limestone decomposing.	1 to 5 per cent across small[a] aggregate. Cylinder expanding, cracking on outside.	Heating decomposed cement; limestone starting to decompose at 700 C.
Sandstone	150 to 500	Strong; quenching at 500 C yields fine mortar cracks.	5 per cent across small[a] aggregate.	Heating: no visible effect; quench: fine cracks at 500 C.
	600, 700, 800 (See Fig. 9)	Weakened; aggregate clean on surface; fine mortar cracks.	2 to 5 per cent across weaker particles. 700 C; expanded, cracked cylinders.	Heating breaks down cement. Note: quartz $\alpha \rightarrow \beta$ at 573 C weakens.
Expanded slag	200 to 600	Strong; contact cracks, clean surfaces at 600 C (weakened).	15 to 20 per cent across small[a] aggregate. Follows large aggregate surfaces.	Heating: no visible effect; quench: fine cracks at 600 C.
	700, 800 (See Fig. 10)	Weakened, cracking along grain boundaries.	5 per cent across small[a] aggregate. Inter-aggregate cracks, powdery cement.	Heating decomposes cement.

[a] NOTE.—"Small" aggregate refers to particles $\frac{1}{4}$ to $\frac{1}{2}$ in. in diameter.

ASTM Standard Method C 116 – 49,[6] the modified cube method.

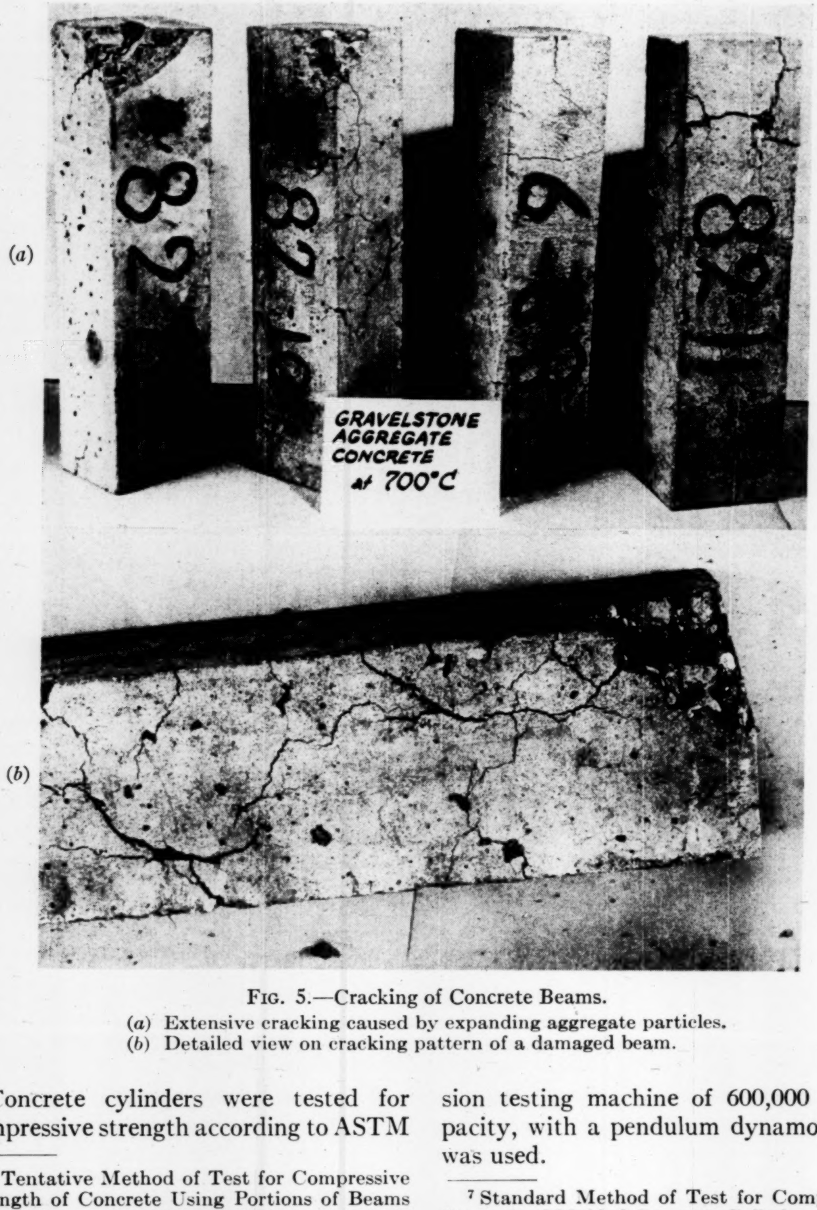


FIG. 5.—Cracking of Concrete Beams.
(a) Extensive cracking caused by expanding aggregate particles.
(b) Detailed view on cracking pattern of a damaged beam.

Concrete cylinders were tested for compressive strength according to ASTM Standard Method C 39 – 56 T.[7] For all compression tests, an Amsler compression testing machine of 600,000 lb capacity, with a pendulum dynamometer, was used.

---

[6] Tentative Method of Test for Compressive Strength of Concrete Using Portions of Beams Broken in Flexure (Modified Cube Method) (C 116 – 59 T), 1959 Supplement to Book of ASTM Standards, Part 4, p. 653.

[7] Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 59), 1959 Supplement to Book of ASTM Standards, Part 4, p. 655.

To compare thermal properties of concretes prepared with different types of aggregates, the following tests were carried out:

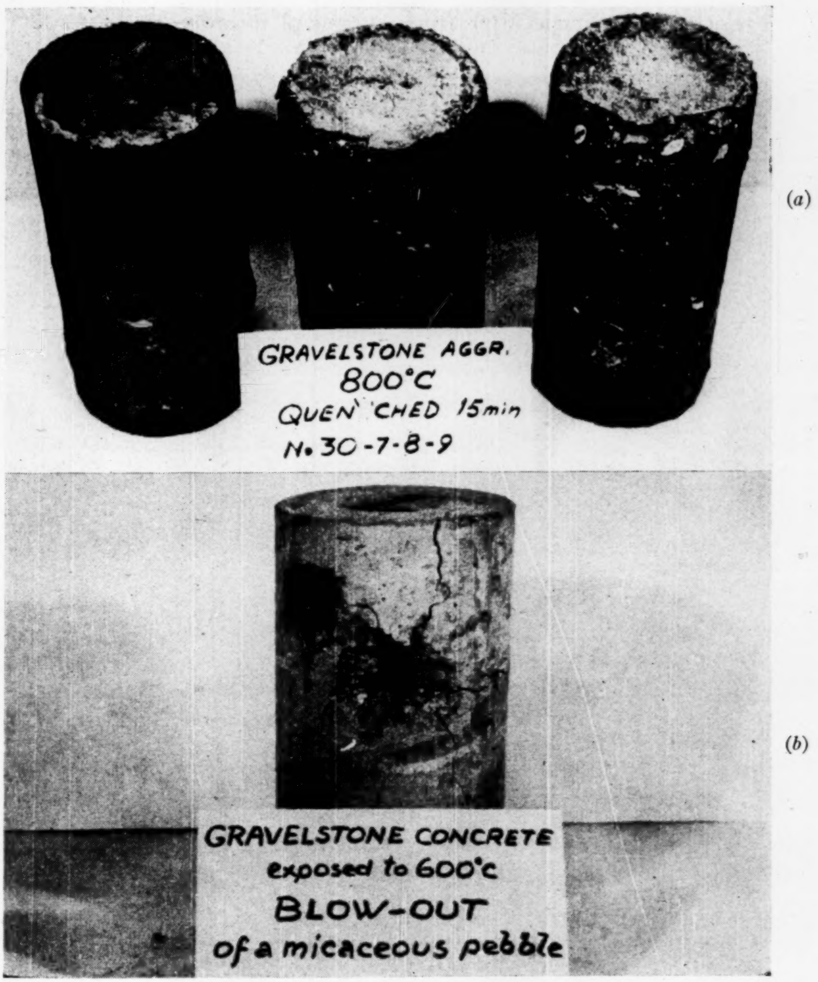

GRAVELSTONE AGGR.
800°C
QUEN'CHED 15min
N.30-7-8-9
(a)
GRAVELSTONE CONCRETE
exposed to 600°c
BLOW-OUT
of a micaceous pebble
(b)

FIG. 6.—Cracked and Disintegrated Concrete Cylinders.

(*a*) Retaining only 15 per cent of original strength.
(*b*) Radial cracks and "pop-out" caused by a micaceous pebble.

1. Thermal conductivity was determined on 3 by 13 by $17\frac{1}{2}$ in. concrete slabs in accordance with ASTM Standard Method C 202 – 47.[8]

2. Specific heat values of $1\frac{1}{2}$ by $1\frac{1}{2}$ by 4-in. concrete specimens were determined by the method of mixture, in a calorimeter using water.

PHYSICAL TEST RESULTS

Test results on concrete after exposure to various elevated temperatures are shown in Table VIII. They are based on average values from tests on either three cylinders or four beam specimens.

---

[8] Standard Method of Test for Thermal Conductivity of Fireclay Refractories (C 202 – 47), 1958 Book of ASTM Standards, Part 5, p. 289.

Pulse velocity of specimens after firing is shown as a percentage of that before firing. No measurements of pulse velocity were made on concrete beams prepared with expanded slag aggregate, because specimen surface porosity caused inconsistency in the test results.

Fired flexural and compressive strengths of test specimens are shown as a percentage of the strength values of companion control specimens.

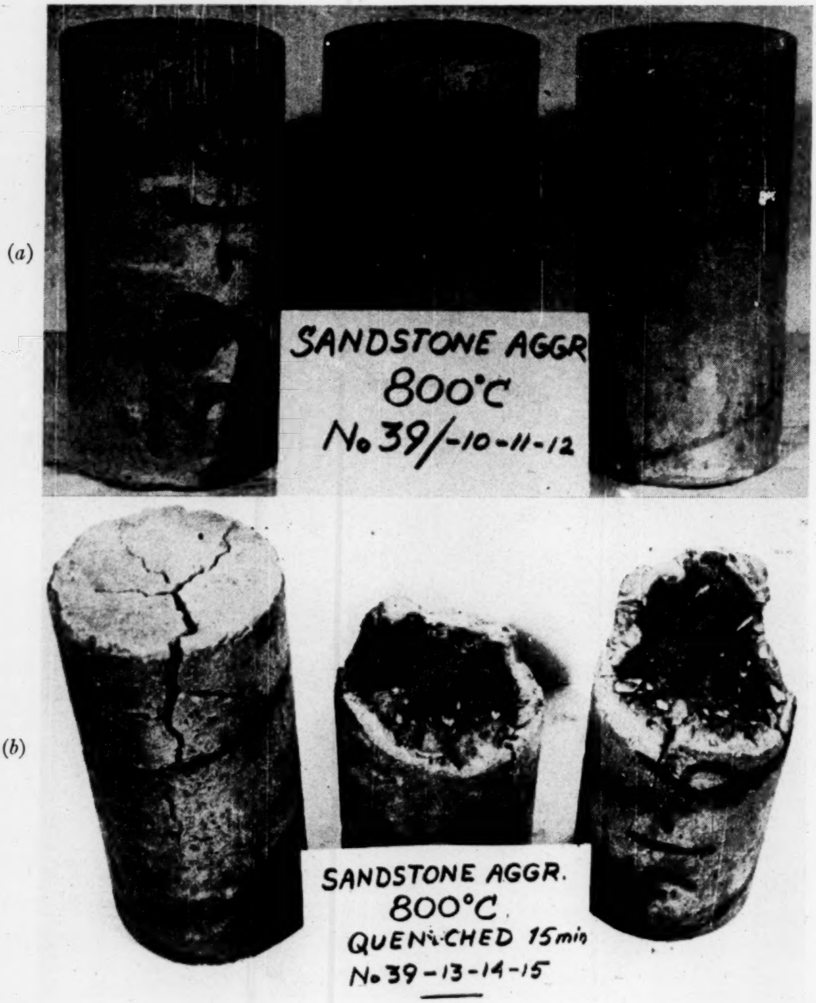


FIG. 7.—Cracked Concrete Cylinders.

(a) Finely cracked, extremely weak—some broke during handling.
(b) Rim of hardened concrete produced by rehydration of cement during quenching.

Weight loss of the room-dried specimens, after heat exposure, is shown in Table IX as a percentage of the weight before exposure. The same table shows the permanent length changes, both drying shrinkage and permanent dilation, of concrete specimens determined by direct measurements in units of thousandths of in. per in.

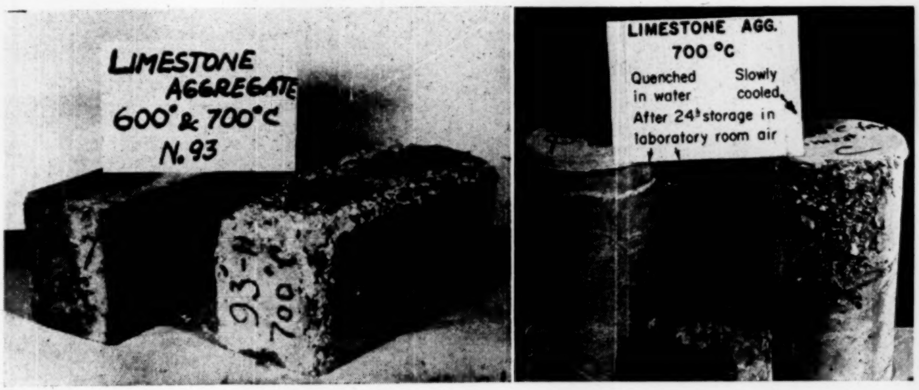
Each figure shown in Table IX is based on the average value obtained in tests on sets of four beams each.

Thermal conductivity and specific heat values for four types of concrete, shown in Table X, were obtained in laboratory tests. The values of heat diffusivity were calculated, using the equation

$$h^2 = K/Cd \quad (5)$$

where:

K = coefficient of thermal conductivity,
C = specific heat value for concrete, and
d = room-dry concrete density.

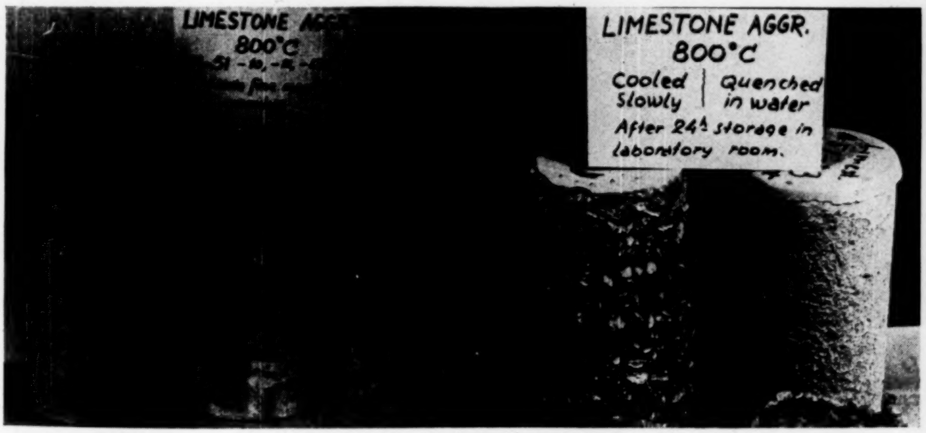

LIMESTONE AGGREGATE 600° & 700°C N.93
93-II 700°C
LIMESTONE AGG. 700 °C
Quenched in water
Slowly cooled
After 24h storage in laboratory room air

(*a*) (*b*)

FIG. 8.—Concrete Beams and Cylinders Showing Effect of Various Cooling Processes.

(*a*) Limestone concrete exposed to 700 C shows spalling caused by slow slaking in air, whereas concrete exposed to 600 C shows no effect.

(*b*) Air-cooled cylinder (to right) is contrasted with quenched cylinder, which shows no spalling by slaking.


LIMESTONE AGG
800°C
LIMESTONE AGGR. 800°C
Cooled Slowly
Quenched in water
After 24h storage in laboratory room.

(*a*) (*b*)

FIG. 9.—Limestone Concrete Cylinders Exposed to 800 C.

(*a*) Unspalled cylinder immediately after removal from furnace.

(*b*) Two cylinders after 24 hr of air storage. Quenching of cylinder (on right) prevented disintegration.

The physical features of heat-exposed and crushed concrete test cylinders are shown in Table XI. Stereomicroscopic examination of the cylinders included studies of aggregate-mortar bond, ruptured particles, and aggregate and cement paste after exposure to elevated temperatures. Some of the heat-exposed test specimens are shown in Figs. 5 to 10. These photographs illustrate dam-

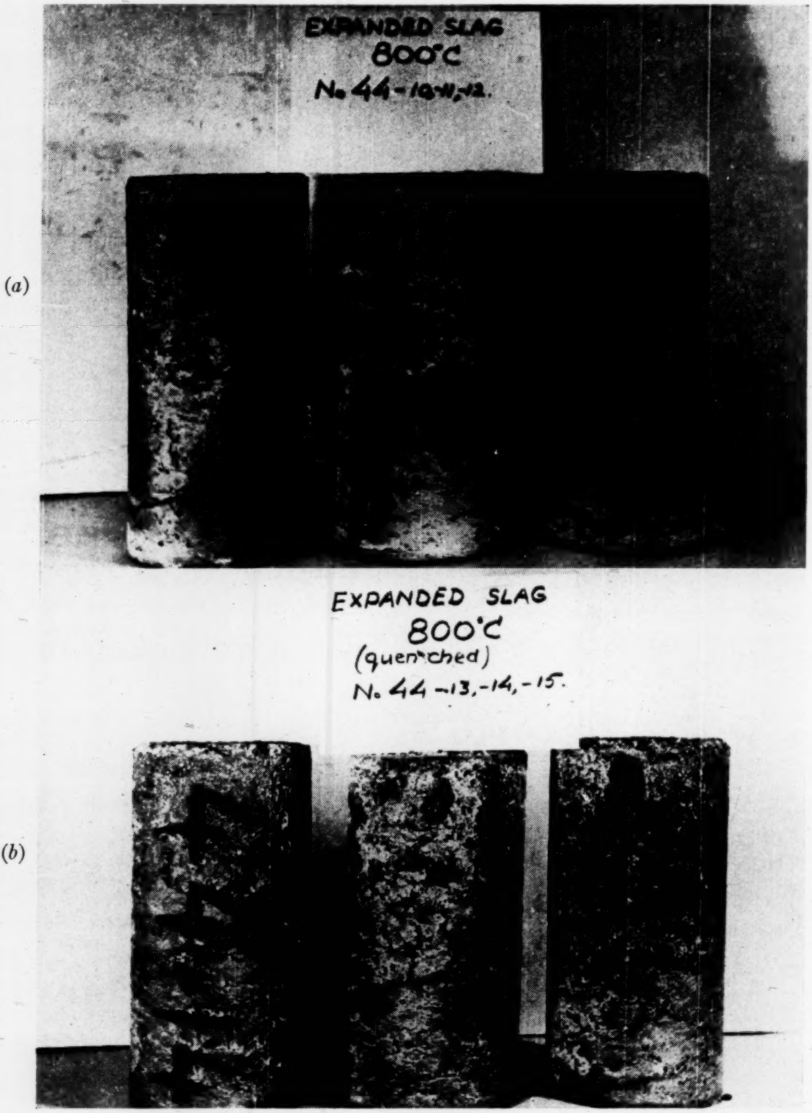

EXPANDED SLAG
800°C
No. 44-10,-11,-12.
(a)
EXPANDED SLAG
800°C
(quenched)
No. 44-13,-14,-15.
(b)

FIG. 10.—Expanded Slag Concrete Cylinders Exposed to 800 C.

(a) Unruptured—dehydrated cement paste has lost its bond with aggregates.

(b) Quenching has rehydrated cement paste, producing a nonflaking skin of sound mortar.

ages sustained by concrete test specimens slowly cooled or quenched after exposure to temperatures of 700 and 800 C.

DISCUSSION OF RESULTS

Beam Specimens:

Results compiled in Table VIII are plotted separately for each type of aggregate in Fig. 11.

Changes in pulse velocity and flexural strength are both represented by smoothly declining curves.

The graphs show that beam-end compressive strengths differ from the flexural strengths, and increase after heating to 200 or 300 C. With further temperature rise, the compressive strength decreases but to a much lesser degree than does the flexural strength.

Gravel and sandstone concretes, after exposure at 500 C, give residual compressive strengths of 71 and 75 per cent respectively of the original, whereas flexural strengths have dropped to 20 and 26 per cent respectively. Limestone concrete performs better, showing in compression 89, and in flexure 42 per cent residual strengths. Expanded slag concrete shows a rapid loss of flexural strength upon heating. It stands up well in compression, showing a sudden loss only after 700 C exposure.

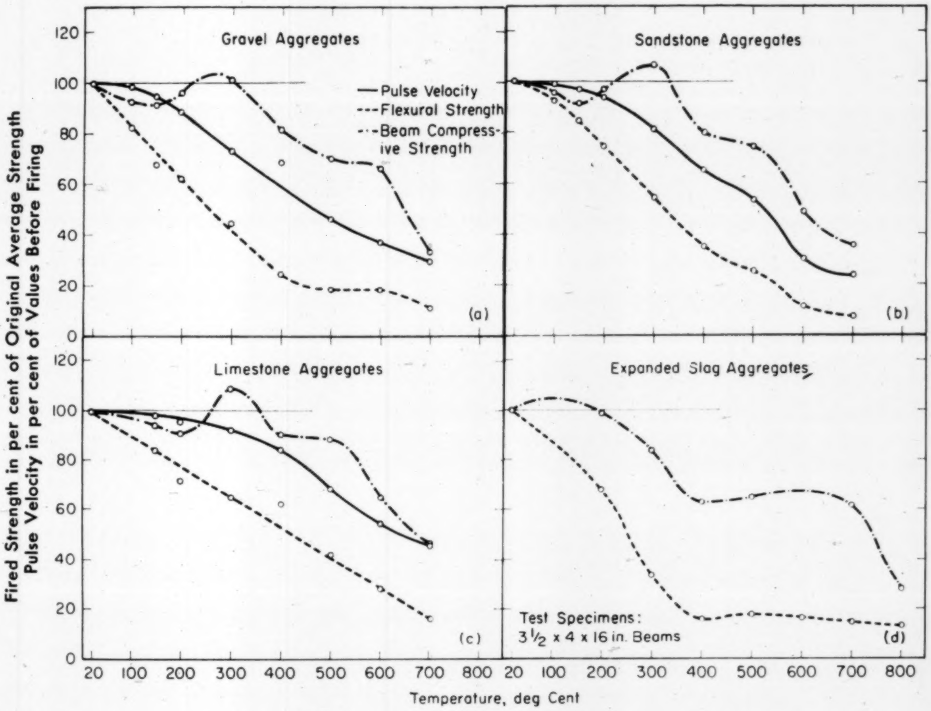


FIG. 11.—Concrete Beams After Fire Exposure.

Cylinder Specimens:

Figure 12 shows graphically the compressive strength of test cylinders.

The solid-line graphs were plotted from the last column in Table VIII and represent the slowly cooled test cylinders. They closely resemble the corresponding curves of the beam-end compression tests.

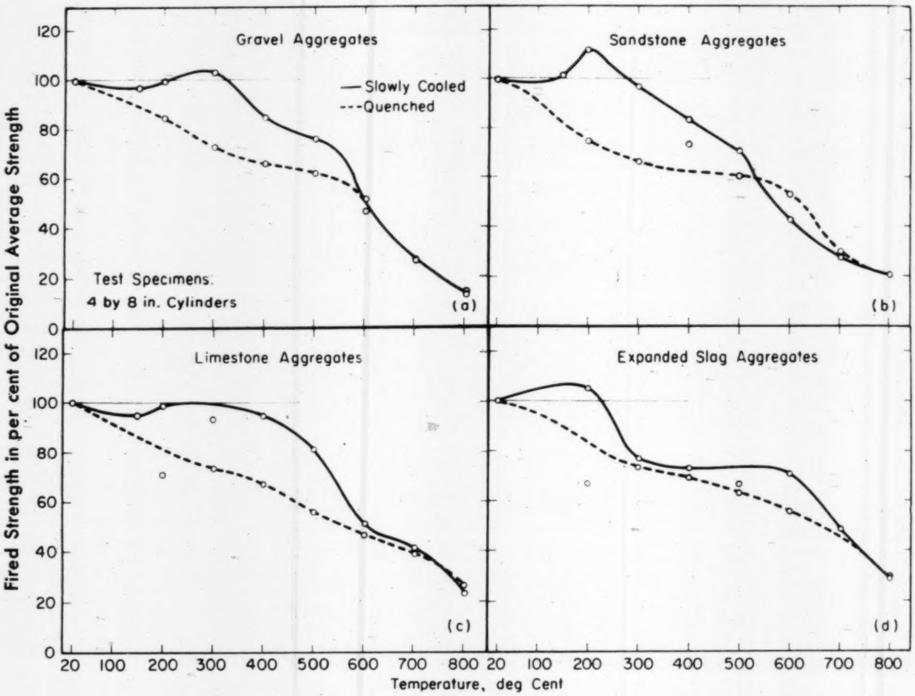


FIG. 12.—Concrete Cylinders After Fire Exposure.

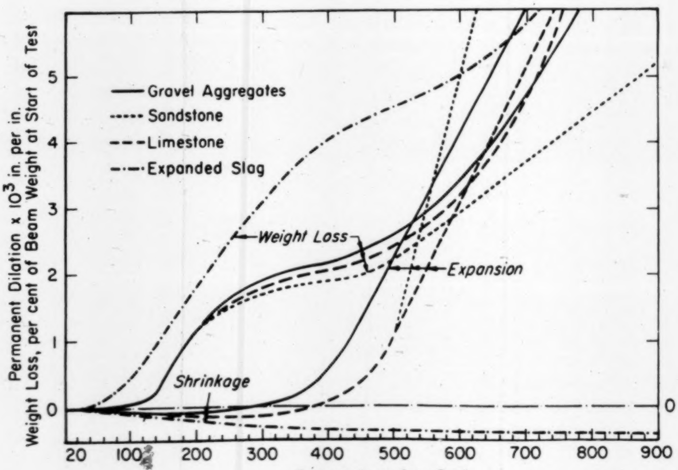


FIG. 13.—Expansion and Weight Loss of Concretes.

The dashed-line graphs represent compressive strength of water-cooled, or quenched, test cylinders. The much lower strength of these test specimens must be attributed to the destructive effect of thermal shock produced by quenching.

The significant drop in strength of the slowly cooled concrete cylinders, after exposure to temperatures above 500 C, is not evident from the tests with quenched cylinders. When broken, the quenched cylinders showed an outer shell of harder, discolored concrete. This shell probably was produced by rehydration of cement by water which penetrated the cylinders during quenching.

From the test results it appears that quenching in water partially restored concrete strength lost by dehydration of portland-cement paste at elevated temperatures (see Figs. 7 to 10).

The curves in Fig. 13 showing permanent length changes and weight losses were plotted from test data compiled in Table IX. Separate curves show weight losses and shrinkage or expansion of concrete specimens made with gravel, sandstone, limestone, and expanded slag aggregates after exposing the concrete to elevated temperatures.

Loss of weight of concrete during heating appears to take place in three stages, as follows:

1. *Drying.*—Evaporation of free moisture by heating from 100 to 400 C.

2. *Dehydration.*—Loss of nonevaporable water, or the water of hydration, by heating from 400 to 600 C (13).

3. *Decomposition.*—Dissociation of calcareous aggregates by release of carbon dioxide in limestone concrete exposed to temperatures over 700 C.

The permanent length changes of concrete specimens after heat exposure resulted from complex physical and chemical changes involving cement paste or aggregates.

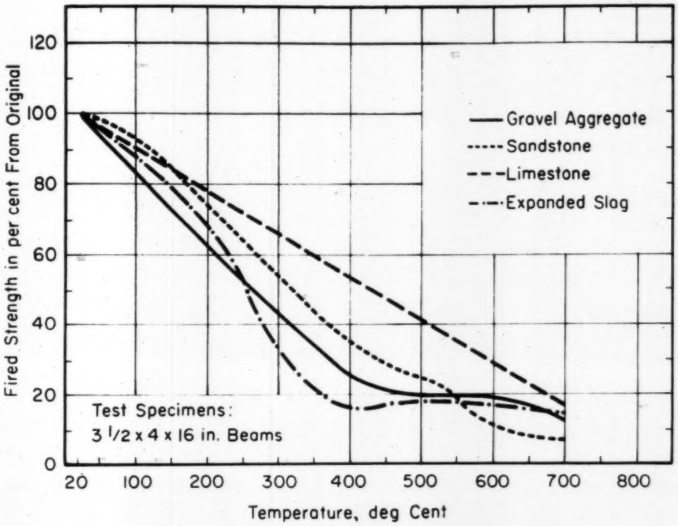


FIG. 14.—Loss of Flexural Strength.

In the lower temperature ranges these changes resulted in a shrinkage but after reaching a temperature above 200 C resulted in a net expansion. A rapid expansion of gravel concrete was observed at 400 C, which was about twice the expansion of limestone concrete. At this temperature the flexural strength of gravel concrete specimens had dropped to 26 per cent of the original. Sandstone concrete showed sudden expansion after exposure to temperatures above 500 C.

Shrinkage of cement paste caused by drying of concrete counteracts thermal expansion. When concrete drying is completed, expansion progresses more rapidly at a rate dependent on the thermal expansion characteristics of the rock type used for aggregate (3).

Concrete made with expanded slag aggregate showed no permanent expansion after heat exposure. A residual shrinkage of 0.03 per cent was observed upon heating to 300 C.

Figure 14 shows losses in flexural strength of concrete made with four different types of aggregates.

According to these curves, the limestone concrete performed best. In the lower temperature range, sandstone concrete had greater strength than other concretes but failed at 530 C. Gravel concrete reaches a low strength at 450 C, which coincides with the rapid expansion of concrete (Fig. 13) and with a drop in thermal conductivity (Table X). The rapid loss in flexural strength of expanded slag concrete between 200 and 400 C may be explained by its higher drying shrinkage, which reached its maximum at 300 C (Fig. 13).

Figure 15 shows changes in ultrasonic pulse velocity on three types of concrete after exposure to various high temperatures.

By means of pulse velocity measurements, the smallest cracks (microcracks) in the cement paste structure can be detected. Such minor internal defects would have little effect on the compressive strength but would be reflected immediately in the flexural strength of concrete.

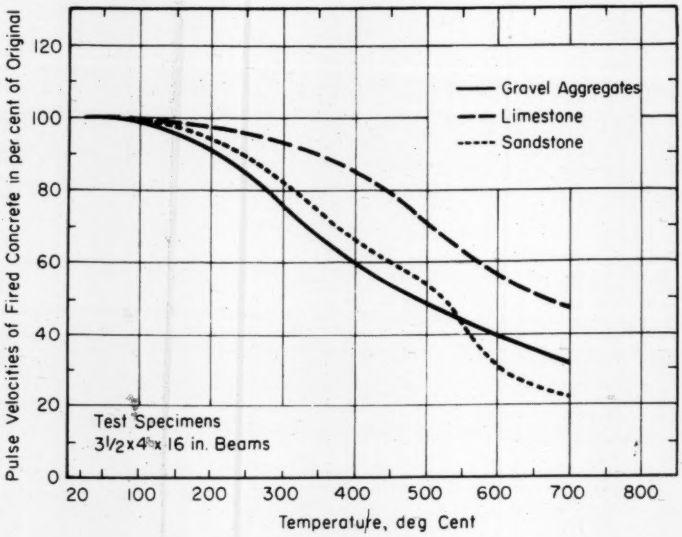


FIG. 15.—Ultrasonic Pulse Velocity.

Changes in ultrasonic pulse velocity reflect changes in structural properties of concrete and its modulus of elasticity (6). The dynamic modulus of elasticity may be computed directly from pulse velocity, V, if the density, d, of concrete is known and the value of Poisson's ratio is assumed 0.24, as follows:

$$E_{\text{dyn}} = 1.83\, dV^2\, 10^{-4} \text{ psi} \quad (7)$$

This equation shows that any changes in structural properties of concrete will affect the dynamic modulus in proportion to the square of the pulse velocity.

A study of Figs. 14 and 15 shows that limestone concrete retains its structural strength at elevated temperatures up to 700 C better than any of the other three concretes investigated.

For temperatures below 500 C, the second best was sandstone concrete. Exposed to temperatures over 500 C, the same concrete showed a rapid loss of strength, which was confirmed also by a sudden drop in pulse velocity. This phenomenon seems to be connected with the inversion of quartz from the α to β form at 573 C (**8**). This inversion causes a volume expansion of about 2.4 per cent, resulting in shattering of crystals (**9**). The rapid expansion of sandstone concrete at this temperature is shown in Fig. 13.

An even more detrimental expansion of sandstone concrete may be expected at 870 C, when quartz inverts to tridymite with an accompanying volume increase of 14 per cent (**9**).

Figure 16 shows changes in compressive strength in cylinders.

In general, the results indicated that after heating to 200 or 300 C the residual compressive strength of concrete increased up to 10 per cent over the original strength. This may be explained partly by heat-stimulated cement hydration with water, still available in the room-dried test specimen, and partly by the densifying process of cement gel due to the loss of physically adsorbed water (**10**).

The strength increase was more pronounced in concrete made with sandstone aggregate. Apparently some free lime was still available in the cement paste to combine at higher temperatures with finely-divided silica in sandstone aggregate, thereby adding strength to the cement paste (**11**,**12**).

Strength dropped rapidly after exposures above 500 C, due to the dehydration of cement paste (**13**). Deterioration effect was observed first in concretes of higher thermal diffusivity (heat penetrating properties). Expanded slag concrete, the heat diffusivity of which is only half that of sandstone concrete, be-

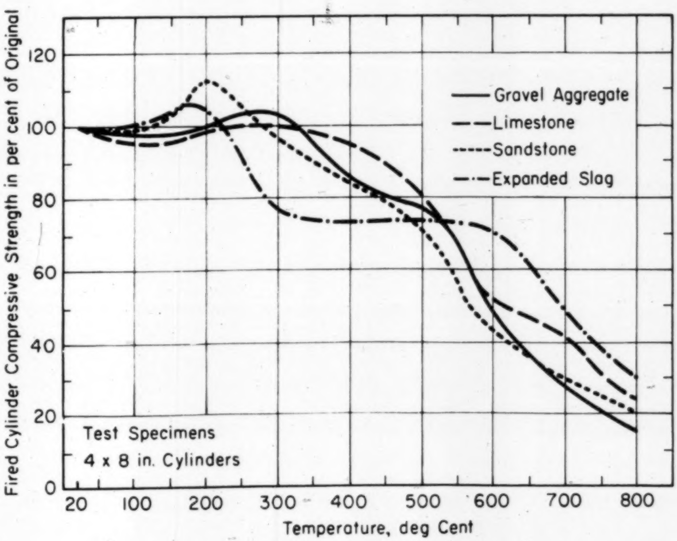


FIG. 16.—Loss of Cylinder Compressive Strength.

gan to disintegrate at a furnace temperature about 100 C higher.

CONCLUSIONS

Within the scope of this investigation, it is concluded that portland-cement concrete prepared with commercial aggregates deteriorates on exposure to dry heat at elevated temperatures. Extended deterioration of concrete is dependent to a large degree on the type of the aggregate.

Results showed evidence that flexural strength is more seriously affected by such exposure than compressive strength.

The effect of different aggregates on the relative fire resistance of concrete may be summarized as follows:

1. Concrete made with gravel, consisting predominantly of crystalline igneous and metamorphic rock, deteriorated more rapidly than limestone concrete. After 400 C exposure, the residual flexural strength of gravel concrete was only 26 per cent and the compressive strength only 85 per cent of the original strength.

2. Sandstone concrete showed a significant compressive strength gain in the lower temperature ranges. Above 500 C it deteriorated, losing strength rapidly.

3. Limestone concrete performed best of the four concretes investigated, after being exposed to temperatures up to 700 C.

4. Expanded slag concrete was strong in compression, retaining 71 per cent of its original strength after exposure at 600 C, but it retained only 16 per cent of its flexural strength after exposure to 400 C.

REFERENCES

(1) "Fire Resistance Ratings," National Board of Fire Underwriters, New York, N. Y., Jan. 1960.

(2) C. C. Carlson, "Function of New PCA Fire Research Laboratory," Journal of Research and Development Laboratories, Portland Cement Association, Vol. 1, No. 2, May 1959, pp. 2–13.

(3) Robert Philleo, "Some Physical Properties of Concrete at High Temperatures," *Bulletin 97*, Research Department, Portland Cement Assn., Chicago, Ill., Oct. 1958.

(4) J. C. Saemann and G. W. Washa, "Variation of Mortar and Concrete Properties with Temperature," *Journal*, Am. Concrete Inst., Nov. 1957, pp. 385–395.

(5) "Thermal Properties of Concrete," U. S. Bureau of Reclamation, Boulder Canyon Project, Final Report, Part VII, Bulletin 1, Denver, Colo. (1940).

(6) R. Jones and E. N. Gatfield, "Testing Concrete by an Ultrasonic Pulse Technique," *Technical Paper No. 34*, Dept. of Scientific and Industrial Research (England), Road Research Laboratory (1955).

(7) J. R. Leslie, "Proposed Tentative Method of Test for the Measurement of the Pulse Velocity of Propagation of Elastic Waves, in Concrete," ASTM BULLETIN, No. 204 Feb. 1955, pp. 19–21.

(8) A. E. Dodd, "The Forms of Silica," *Ceramics*, Symposium, British Ceramic Soc. (1953).

(9) W. Ahrens and J. H. Hellmers, "Rohstofforschungen feuerfester Quarzite," *Die Technik* (Berlin), Vol. 2, No. 9, Sept. 1947.

(10) H. Busch, "Feuereinwirkung auf nicht brennbare Baustoffe und Baukonstruktionen," Zementverlag, GmbH, Berlin (1938).

(11) R. E. Stradling and F. L. Brady, "Fire Resistant Construction," *Special Report No. 8*, Dept. of Scientific and Industrial Research (England), Building Research Board, London (1927).

(12) G. L. Kalousek, "The Reactions of Cement Hydration at Elevated Temperatures," *Proceedings*, International Symposium on the Chemistry of Cement, London (1952).

(13) J. S. Blackman, "Method for Estimating Water Content of Concrete at the Time of Hardening," *Journal*, Am. Concrete Inst., March 1954, pp. 533–541.

OBSERVED SETTLEMENT OF PRELOADED SOFT, FINE-GRAINED SOILS*

By E. J. Zegarra[1]

Synopsis

Data are presented in this paper indicating that the field rate of settlement is faster than predicted by laboratory tests. One implication of the results is that preloading, usually an economical method, is capable of effecting even larger savings by advancing completion time. Observations of settlements on three preloaded areas underlaid by soft, clay soils of recent origin and, in two cases, of high organic content show that the reaction of the deposits is almost simultaneous with loading. As an explanation of the phenomenon, it is suggested that the processes of sampling and specimen preparation decrease the permeability of a thin surface layer. It is concluded that the computed time for a given settlement be regarded as the possible limit rather than the probable and practical time experienced in the field.

In the construction of three recent projects where preloading was used to improve the supporting capacities of areas underlaid by soft, compressible, fine-grained soils, it was found that a given degree of consolidation was reached in a time much shorter than that estimated from laboratory tests. The savings accrued by this shorter time were added to the economies effected by the preloading method. Other methods considered were (1) use of piles, (2) removal of soft soils, and (3) expenditure of considerable sums of money in maintenance to remedy the effects of long-time subsidence. As the weak soils were below water, excavation and replacement would have been impractical, and, in the case of a large storage tank, the cost of piling would have been equal to the cost of the tank itself. Common to the three projects were the recent origin and attendant soft consistency of the soils. In two locations, the fine-grained soils were highly charged with organic matter.

It is believed that in laboratory test specimens, which are disturbed and smeared at the surface during sampling and trimming, the permeability characteristics are affected sufficiently to make the estimates of consolidation time far in excess of that experienced in the field. This disturbance is most noticeable in organic and stratified deposits.

Preloading is often abandoned in favor of other methods, chiefly because the time required for the consolidation process is not available in the construction schedule. The alternative usually involves considerable additional expense which is justified on the assumption that construction and therefore production are that much closer. However, the original decision based on laboratory test results could be erroneous. In the three cases to be presented, there are indica-

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[1] Soils Engineer, The M. W. Kellogg Co., New York, N. Y.

tions that the time-consolidation relationship established from laboratory tests on organic soils should be regarded as the possible maximum limit for field conditions. A brief review of the preloading method, of subsurface investigations, and of the pertinent aspects of geology and rheology may assist in the interpretation of the results and provide a tentative explanation of the discrepancy between observed and predicted time of consolidation.

REVIEW

Preloading an area with a surcharge approximately equal to the design load prior to construction accomplishes two objectives: first, consolidation of the subsurface soils, thus eliminating or reducing settlement under the load of the proposed structure; and second, increase in compressive strength allowing a larger unit load to be supported. Consolidation requires time, which is presumed to exceed construction periods. When schedules permit it, a limited amount of time may be available for preloading. This time can be used to accomplish the objectives of preloading by applying a surcharge heavier than the design load. The magnitude of the surcharge is selected so that the consolidation achieved in the time available produces a settlement equal to that predicted for the structure. Doubling the load, however, does not cut time in half, yet a substantial amount of time is saved. Determination of the time required starts with laboratory tests made on undisturbed samples obtained from subsurface borings.

A subsurface investigation achieves two ends: first, the soils are correctly identified with respect to their composition, sequence, and geologic origin; and second, samples of soil are recovered in such condition that the structure and composition remain inviolate, or what may be considered so, using sampling tools of good design and careful operating techniques. Samples for identification of soils may be considered satisfactory only when the structure of the deposit is retained, even if somewhat distorted. Wash and auger samples furnish merely and indication of the type of remolded soil, or of a change in the type of soil, without revealing the character of the deposit. Split tube samples, on the other hand, are, in most cases, satisfactory for examination of the structure and stratigraphy of the deposit, both of which influence the behavior of the stratum under load.

Samples with minimum disturbance are used for laboratory compression tests. But with the best sampling tools, such as the piston and foil samplers, and with the best laboratory techniques disturbances occur at the surface of the material. The metal edge which must shear the soil and cut it to go into the tube smears, spreads, and disturbs the material. And to the extent of this disturbance the soil properties in the region are changed—generally for the worse.

Thorough investigations were made for each of the three projects described, but only the results pertinent to settlements are given here.

STORAGE TANK—VENEZUELA

In the course of planning the development of a tract of land on the north coast of Venezuela for a large industrial project, a storage tank 160 ft in diameter with a unit load of 3000 lb per sq ft was located in an area underlaid by a soft compressible deposit, described as mottled gray to light brown clay with pockets of sand. The dry unit weight ranged from 82 to 105 lb per cu ft with water contents ranging from 37 to 22 per cent respectively. Structurally the soft clay was in a lens or pocket under a superficial, thin layer of beach sand and surrounded by coarse to fine alluvial sand and gravel, and beneath it was a metamorphic rock formation. Figure 1 shows a north-south cross-section of the subsurface soils reconstructed from logs of borings. The recent age of the compressible soil indicated that under the tank load large total and differential settlements would be expected. To avoid this, several schemes were considered: the cost of relocating the tank was estimated at $200,000, piling at $125,000, and removal of the soft clay as impractical as ground water was close to the surface. A construction schedule of two years made it possible to delay the erection of the tank to the final stage of the project, thus making time available for preloading. It was estimated that to preload the area for a period of six months with a fill about 17 ft high would cost $20,000. Laboratory tests predicted settlements of about 12 in. at the end of this period, and residual settlement for the tank fully loaded of no more than 2

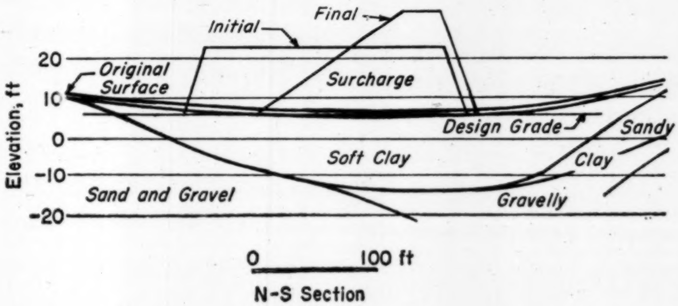

Final
Initial
20
Original Surface
Surcharge
10
Elevation, ft
Design Grade
Sandy
0
Soft Clay
Clay
-10
Sand and Gravel
Gravelly
-20
0
100 ft
N-S Section

FIG. 1.—Soil Profile—Storage Tank, Venezuela.

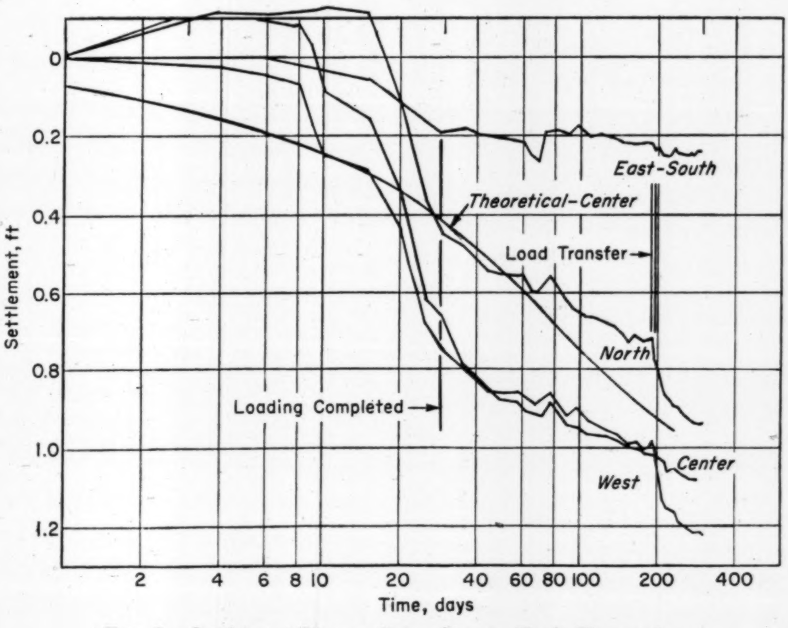


FIG. 2.—Settlement Observations—Storage Tank, Venezuela.

in. at the circumference and 6 in. at the center. To measure settlements, four observation plates were installed 90 deg apart on the periphery and one at the center. Periodic readings were taken and records kept for 290 days.

Figure 2 shows a plot of the observations made as well as a theoretical curve for the center point, constructed from data on a boring at this point. Two features were immediately noted: the northwest quadrant was settling much faster than the other quadrants, and a large percentage of the final settlement occurred in the first 30 days—practically during application of the load. Subsequent settlements proceeded at a diminishing rate, probably as part of secondary consolidation, since the plot shows it as a straight line. As the records indicated that the compressible soil was not as uniform as had been anticipated and as the possibility of future differential settlement could not be ignored, it was decided to transfer some of the surcharge to the sector with larger settlements. Thus the shape of the surcharge took the form of an oblique truncated pyramid with a height of 27 ft. This operation, completed in about five days, induced additional settlements which were registered almost simultaneously with the application of loads. About 3 months later, or 10 months after the original area was loaded, the surcharge was removed and the tank was erected. Examining the plot of observations, some irregularities may be noted, sometimes above and sometimes below the average trend. At first attributed to errors, these variations were found to coincide with changes in load caused by a combination of moisture change in the fill and tidal effects. These small changes in the effective stress were being faithfully and promptly reflected in the observation plates.

WAREHOUSING—DELAWARE

This case involved foundation support of floors for storage of a petrochemical product. As part of a much larger project, there was to be a warehouse building 340 ft long and 200 ft wide. One portion, 140 ft long, was designated to store products in bulk, deposited by overhead conveyors, and creating a triangular loading with a maximum pressure of 1600 lb per sq ft. The other portion, reserved for storage of bagged product, had a loading platform about 4 ft high combining with the bags for a unit load of 700 lb per sq ft.

Fill, mainly granular, 15 ft thick, covering the entire area overlaid a soft compressible deposit of dark gray organic silt varying in thickness from 2 to 15 ft. Originally this was the estuary of the Delaware River with marshland swamp only slightly above sea level. Formed in quiet waters and with rich vegetative cover, the soils were soft and highly charged with organic matter. The organic content of this soil, although undetermined, may be judged from the dry unit weight which ranged from 40 to 70 lb per cu ft. Under this organic silt was a layer of clayey silt much less organic, slightly firmer and generally thin. Next, below, was a fairly thick stratum of dense sand and gravel deposited at the time of the glaciers by alluvial and fluvial agents. At a depth of 40 ft or more below the present surface are the metamorphic rocks—schists and gneisses—of the province.

The organic silt and the clayey silt have been consolidated and stabilized by the pressure of fill, but the additional warehouse loads would again stress these weak soils and renew the process of consolidation and settlement. A longitudinal section through the building is shown on Fig. 3 illustrating the subsoil conditions as disclosed by borings. With the thickness of the compressible soils varying over the site, differential settlements were anticipated. To eliminate or reduce settlements, the floor could be supported, either on piles, as the walls were, at a cost of $175,000 or on soil after preloading, at a cost of $20,000. The height of required fill was determined by the loads and thickness of compressible soil at a given point. Since the time available for preloading could not exceed six months, the height of the fill was increased, thus loading the soil to higher than design pressures. Estimates of ultimate settlement were made assuming simultaneous compression from primary and secondary consolidation. Twelve observation plates were installed at strategic locations. Some of these are shown encircled and numbered in Fig. 3. Loading was completed in about 30 days by a fleet of

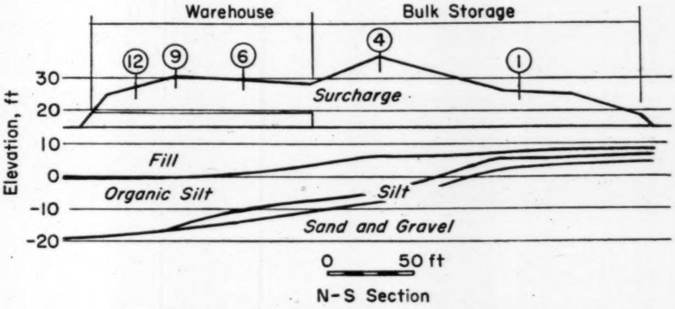

Warehouse
Bulk Storage
⑫
⑨
⑥
④
①
Surcharge
Fill
Organic Silt
Silt
Sand and Gravel
Elevation, ft
30
20
10
0
-10
-20
0
50 ft
N-S Section

FIG. 3.—Soil Profile—Warehouses, Delaware.

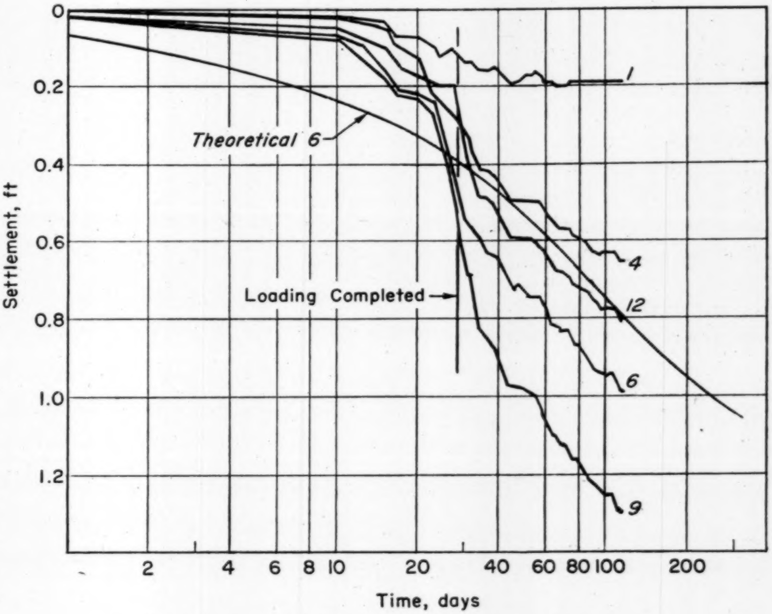


FIG. 4.—Settlement Observations—Warehouses, Delaware.

trucks and a few bulldozers spreading and shaping the fill. Figure 4 shows a plot of the observations made on five of the twelve plates. At the start, and for the first 10 days or so, fill placement was slow but was accelerated between the 15th and 25th days. Similar behavior was observed in the settlement plates: while at the beginning the settlement was quite small, it increased to 55 per cent of the final values between the 20th and 30th days. Thereafter and to the end of the observations, the graphs tend to flatten, indicating the steadily diminishing rate of settlement from secondary consolidation. At the end of 120 days, when construction was underway, the observations were discontinued owing to accidental damages to the plate assemblies. Up to that time, the ratio between predicted and observed settlement ranged from 0.60 for plate 4 to 0.88 for plate 9 where the largest movement took place. A theoretical curve is shown for point 6 computed from tests made on a nearby boring. The comparison of the theoretical and field curves shows the difference in rate of settlement, the speed with which primary consolidation occurs, and the significance of secondary consolidation.

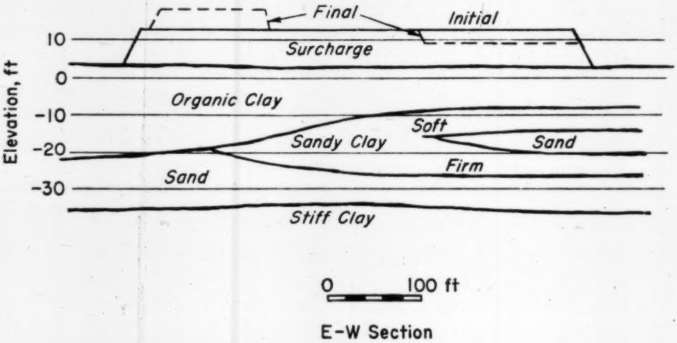


FIG. 5.—Soil Profile—Texas Site.

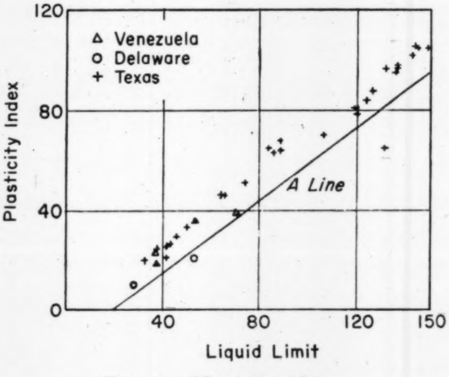
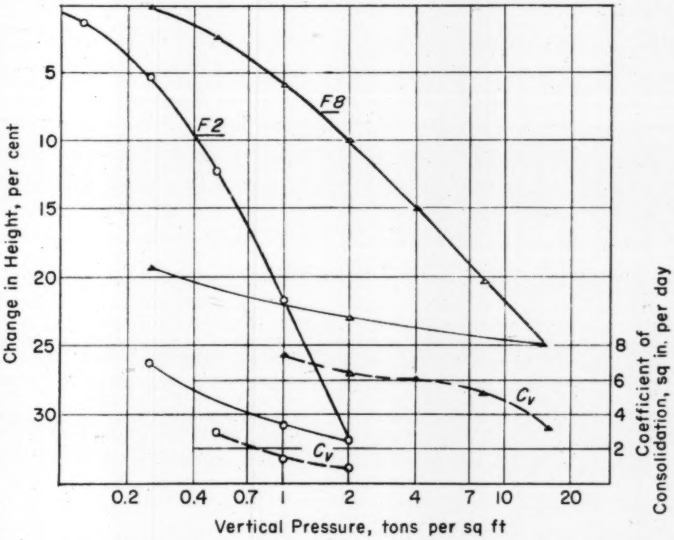


FIG. 6.—Plasticity Chart.

SITE PREPARATION—TEXAS

An area 500 ft square selected as the site for additional facilities in an existing plant was preloaded, not to support equipment loads but merely to carry an additional fill 1 ft thick above the existing surface. This site was a marshy swamp along the banks of the Neches River, near the Gulf of Mexico. Subsurface investigations disclosed that the upper stratum was a very soft organic clay incapable of supporting loads without settlement. This deposit, varying from 15 to 24 ft in thickness, was underlaid by an alluvial deposit of fine to coarse quartz sand with fine, polished gravel. The combined thickness of the two strata varied little from 37 ft. At this depth was found the stiff, slickensided, Pleistocene clay common to southeast Texas. Ground elevation was 2.5 ft above mean low gulf level, and water was at the ground surface. A typical section of the subsurface in an east-west direction is shown on Fig. 5. The organic clay deposit contains decayed and decomposed vegetal matter ranging from finely divided to roots and even logs. Near the bottom it is less organic, more sandy, and becomes a little stronger. Figure 6, plasticity chart, shows the results of tests on this soil. Liquid limits up to 60 per cent belong to the soils near the bottom of the stratum while the upper, organic clays had values up to 240 per cent. In most cases, the natural water content was near the liquid limit. Consolidation tests for two typical samples are shown on Fig. 7, one for the sandy clay with 3 per cent of organic matter and the other for the soft clay with 13 per cent. Organic contents up to 24 per cent were measured while the dry unit weight ranged from 35 to 60 lb per cu ft.



Boring	F2	F8
Unit dry weight	37	96 lb per cu ft
Water content	124	26 per cent
Liquid limit	131	35
Plastic limit	66	14
Void ratio	3.23	0.72
Specific gravity	2.49	2.67
Organic content	13	3 per cent

FIG. 7.—Consolidation Tests—Texas.

All equipment on this project was supported on piles penetrating to the Pleistocene clay; all underground pipe lines and utilities were supported on the clay. Pavement grade for the entire site was 1 ft above natural grade. Fill placed for grading would cause subsidence and damage to the underground piping, exert drag on the piles and break up pavements and roads. To minimize these dangers, the site was preloaded with 10 ft of fill for six months. It was estimated that after this time the remaining settlement would not exceed 0.2 ft. The only alternative to preloading was to burden the plant with constant maintenance repairs for many years.

Fill was placed in about 20 days by a hydraulic dredge pumping 92,000 cu yd of clean sand from a nearby channel at a cost of $36,000. To observe settlements, 12 plates were installed at the ground surface, and after the fill was completed, four piezometers were constructed with tips halfway into the organic clay. These observation points were located where borings had been made. Figure 8 shows the range of settlement for typical plates and a theoretical curve computed from tests and modified from experience in these soils. Notable in these observations is the immediate response to load: large during placement and at a diminishing rate after completion. In the west portion of the site, where the clay was thicker and softer, the settlement was larger than elsewhere but no different in total time. After 140 days, when the predicted settlements were nearly complete, some of the fill was transferred from the east to the west side in order to induce further consolidation and reduce possible future settlement. Five feet of fill was added in about 10 days. Again the response was immediate, and, after completion of the transfer, settlements resumed at a decreasing rate. Thirty days later the surcharge was ready for removal. Observations made in three typical piezometers, shown on Fig. 9, indicate a fairly uniform rate of pressure dissipation. How quickly changes in stress were reflected in the pore water pressure may be appreciated

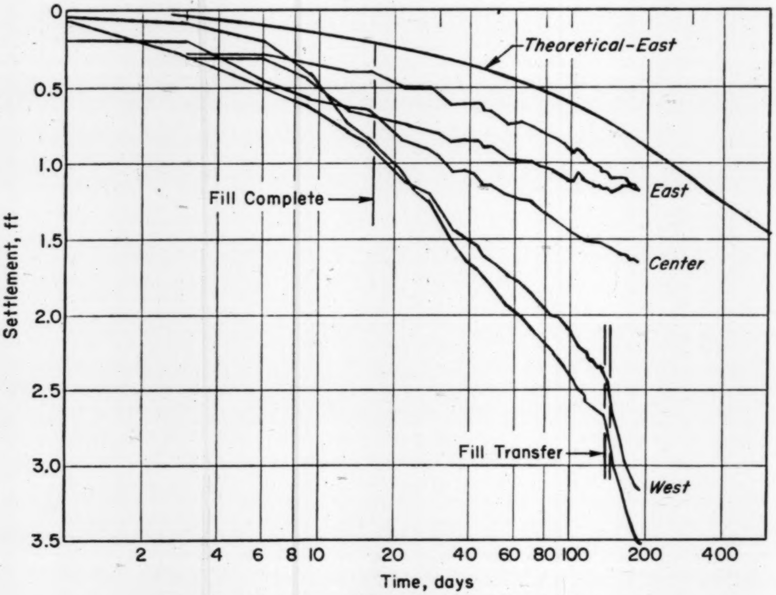


FIG. 8.—Settlement Observations—Texas Site.

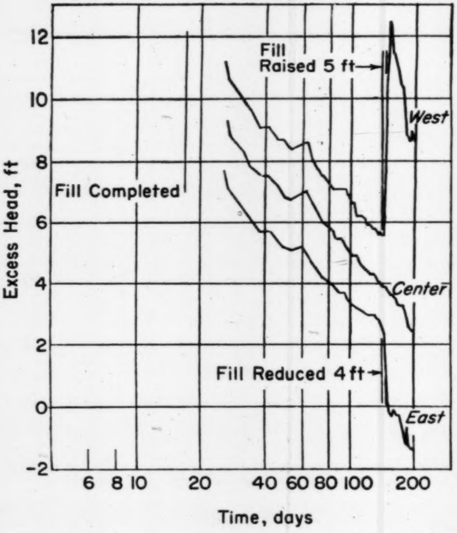


FIG. 9.—Piezometer Observations—Texas Site.

by the sudden drop and rise of water level at the time the transfer of load occurred. The drop of level below zero datum at the east side represents the effect of certain drainage measures required for construction.

COMMENT ON RESULTS

In the three examples the reactions to change of stress observed in the field showed discrepancies with the predictions from laboratory tests. If the estimated magnitude of settlement is accepted as being correct, although the field records seem to include secondary compression effects, the time for primary consolidation is shorter than predicted. It is this feature of the examples that affords most practical interest and invites speculation.

Consolidation test data for these soils exhibit the familiar characteristics of inorganic clays with sharp distinctions between primary and secondary consolidation. From these tests, the only indication revealing the softness and hinting at the high organic content of the soil is the shape and large volume change shown in the pressure-compression curves. But the noteworthy feature is the small and easily reduced coefficient of consolidation by which the rate of settlement is computed, thus indicating an abnormally small coefficient of permeability, or nearly impervious soils. The test results were therefore modified to agree with experience in the case of soils in Texas. Also of interest in the tests is the position of the clays in the plasticity chart: they are located well above the A line in the region reserved for inorganic soils. These values probably represent the plasticity characteristics of the mineral matrix unaffected by the admixture of organic matter which is characteristic of young deposits and is in accordance with results for similar soils in Texas.

Few, if any, are the soils deposited in conditions so uniform that stratification is totally absent. Most soils show stratification and thereby variations in grain size and permeability. In the case of highly organic soils, the remains and fragments of vegetal matter possess voids and channels much larger than those of the surrounding mineral matrix. Water flows freely through these channels when pressure is applied, as it does through the openings of coarse-grained soils. But these structures are changed when the soil is sampled and a specimen is prepared.

A consolidation specimen is trimmed using knives, saws, wires, or spatulas after it is extracted from the tube containing the larger sample. It is an inevitable consequence of the processes of sampling and trimming to erase and smear the surfaces and so to alter the original structure. The continuity of the pores through which water flows is abruptly obstructed. To restore flow, new drainage paths must be open; voids must be filled; links reestablished with the inner structure; and, if pressure is then applied, water flow is restricted requiring longer time to drain. This can only occur when the permeability of the remolded zone is smaller than that of the intact material. Evidence of fast drainage was shown in the cases of Venezuela and Texas when shifting of loads was immediately reflected in settlements, and even piezometers reacted without significant lag. However, the changes in the magnitude of pressure head following the load transfer indicate that disturbance at the surface of the piezometer wells reduced the permeability of the soil. Another factor of importance is the lateral drainage which takes place in the field when stratified or organic soils are involved. This, of course, is not included in any laboratory test and, unfortunately, impossible to measure with existent equipment. But were it possible to do so—for example, by the use of porous rings—the consolidation theory would need to be expanded into the three-dimensional frame.

CONCLUSIONS

As a result of settlement observations made in three cases involving organic clay, organic silt, and soft clay of recent origin the following conclusions are advanced:

1. Test specimens of soft, compressible clays or silts, especially when they have high organic content, are adversely affected by sampling and trimming.
2. Cutting and trimming samples for test specimens disturb the surface and decrease the permeability characteristics of the soil.
3. Time rate of settlement estimated from laboratory tests is longer than field performance demonstrated in the cases cited.
4. As the surface disturbance has large but indeterminate effects, the computed time of consolidation ought to be regarded as the maximum or limiting for field performance.

Acknowledgments:

Subsurface investigations and studies were made by Dames & Moore, Soil Mechanics Engineers, New York, N. Y., for the projects in Venezuela and Delaware. McClelland Engineers, Houston, Tex., performed similar services for the Texas project. The M. W. Kellogg Co. as designer and builder of these projects was involved in the decisions of preloading, recording, and evaluation of results.

DISCUSSION

MR. GARDNER M. REYNOLDS[1] (*presented in written form*).—The writer was directly involved in two of the projects described by Mr. Zegarra: the one in Venezuela and the one in Delaware. In both cases, our firm was retained to perform foundation investigations and develop recommendations for foundation design.

Storage Tank—Venezuela:

At the time the test borings were drilled in Venezuela, definite locations of structures and equipment had not been determined. Borings were drilled on a grid pattern throughout the site, with concentrations in areas where heavy foundation loads were expected.

Several months after completion of the field work, it was decided to locate the storage tank described by Mr. Zegarra in an area underlaid by soft soils. Fortunately, one boring had been drilled within the area to be occupied by this tank. In addition, a second boring had been drilled some 30 ft outside the location of the tank periphery. The data obtained from these two borings were used to develop the surcharge design and procedure.

The borings were drilled with a rotary-wash boring rig, and driller's mud was used to prevent caving of the boring walls. Samples of the soils were extracted using a Dames & Moore soil sampler. Where possible, the soil sampler was pressed into the soil using the drill rig as a reaction. Where this could not be done, the samples were obtained by driving the sampler with a 500-lb weight falling 24 in.

As shown on the author's Fig. 1, the soils under the tankage area consisted of some 15 to 20 ft of relatively soft clay underlaid by sand with gravel or sandy clay. A number of the samples of soft clay were subjected to unconfined compression and triaxial tests to determine their strength characteristics, and one sample was subjected to a consolidation test. Had we been able to anticipate that these data would be needed to develop surcharge design criteria, we would have performed additional consolidation tests to define more accurately the physical properties of the compressible soils. However, as it developed, we were forced to use the data that had been accumulated.

---

[1] Partner, Dames & Moore, New York, N. Y.

In designing the surcharge and preparing the area for construction of the tank, consideration was given to two problems. It was necessary to induce sufficient consolidation of the soft soils so that residual settlement after the tank was in operation would not be detrimental, and it was necessary to be sure that a shear failure would not occur when the soils were subjected to the stresses imposed by the loaded tank. In order to determine the increase in strength under full consolidation, it was assumed that the gain in strength under full consolidation would be equal to the imposed surcharge load times the tangent of 16.5 deg, and if full consolidation (100 per cent) did not occur, then the gain in strength would be directly proportional to the percentage of consolidation that did occur under the load imposed for a specified time period.

Having made these assumptions, the amount of total settlement to be expected under the tank without surcharging was computed. The amount of residual settlement after surcharging that the tank could withstand without difficulty was decided upon, and it was concluded that about 1 ft of settlement would be taken out by surcharging for a period of 6 months. A recheck was made to determine if there would be a shear failure after tank erection with the soil conditions that would exist after surcharging.

The consolidation test was performed on a sample 1 in. in height and approximately $2\frac{1}{2}$ in. in diameter. The sample was in its original container rings which are within the soil sampler that was used. Hence, there was no extrusion, paring, and reconfinement in the laboratory. There is no doubt, however, that there was smearing of the sample surfaces in order to prepare it for the consolidation machine.

The rate of primary consolidation was computed using the square-root-of-time method.

Warehousing—Delaware:

The problem at Delaware was somewhat different. In Delaware, it was considered necessary to design the surcharge so that only minor residual settlements would occur after construction of the building. In addition, sufficient subsurface data were available so that it was known that the conditions under the structure were nonuniform; also, the loads to be imposed varied in the building in both the longitudinal and transverse directions. Construction time was limited; 6 months was the maximum time that could be allowed to achieve the necessary preconsolidation. Boring procedures and laboratory tests were similar to those described for the Venezuelan project. Through an analysis of the laboratory consolidation test data, it was determined that the full consolidation due to the design loading could not be achieved in 6 months unless a higher load was applied in some areas than that which would be applied after construction. Figure 3 of the author's paper shows how the surcharge load was proportioned to take into account the variables, namely, design load after construction, difference in compressible strata thickness, and differences in compressibility characteristics of the compressible soils.

In comparing the field settlements in both Venezuela and Delaware with the computed settlements, the shapes of the curves are significant. It is evident that a relatively high percentage of the computed settlement occurs more rapidly than would be predicted, but that the total anticipated settlement for 6 months seems to occur in about the predicted time.

On the Delaware project, it had been predicted that the following settlements would occur after a surcharge load had been applied for 6 months at the points shown on the author's Fig. 4:

Point 1	4 in.
Point 4	16 in.
Point 6	14 in.
Point 9	18 in.
Point 12	14 in.

By extrapolating the observed settlements out to 200 days (about 6 months after completion of the placement of surcharge) on Fig. 4 of the author's paper, the following approximate settlements are obtained:

Point 1	2.4 in.
Point 4	8.9 in.
Point 6	14.0 in.
Point 9	18.2 in.
Point 12	11.4 in.

It is believed that the correlation between total anticipated and observed settlements in both cases was reasonably good, taking into consideration the possible variations in subsurface conditions not disclosed by the borings. If the predictions had been made for a 3-month period, considerably more settlement would have occurred in this period than predicted. Generally, it then would appear in the cases observed that primary consolidation occurs more rapidly in the field than in the laboratory. However, it is believed that secondary consolidation also occurs in the laboratory. In computing total settlements and time rates, we may be introducing compensating errors. The computed settlement based on laboratory tests contains some settlement due to secondary time effects and is probably quite close to the total settlement that would occur in the field. The computed time rate for primary consolidation is probably too slow for the reasons pointed out by the author; however, if the preloading time is arbitrarily reduced because of the errors in testing, then the residual settlement after construction may be too severe.

It is believed that more experience records on these conditions are needed before final conclusions can be made.

MR. J. A. FOCHT, JR.[2] (*by letter*).—Presentation of performance records on these three successful preload fills provides a basis for encouraging the use of this technique of site preparation. It is significant to note that in two of the cases reported, changes in fill thickness were made during the preload period on the basis of settlement observations. Continuous review of settlement and piezometer data during any preloading operation, as was obviously done for the three projects reported, is one key to successful achievement of the desired purpose. A very encouraging conclusion of this paper is that the time-rate of consolidation under a preload fill will be faster than would be normally predicted on the basis of laboratory tests.

The comparison of predicted time-settlement rates with actual observations for the three cases adds materially to the very limited amount of field data published on time-rate of settlement. The author concludes that the field rate of settlement is faster than that predicted from laboratory rate because of unavoidable sample disturbance inherent in sampling and test preparation. Sample disturbance undoubtedly can, and probably did in the studies cited, have a significant effect on the laboratory time-consolidation curves, which would result in an overprediction of time required for field consolidation. The two purposes of this discussion are, however, to point out that for the Texas site (1) the difference between the field time-consolidation curves as observed and those predicted from laboratory tests is as much in the shape of the time-consolidation curve as in the time to full consolidation, and (2) the piezometers indicated dissipation of excess pore pressure equal to 90 per cent primary consolidation when the settlement plates indicated only about 30 per cent total consolidation.

---

[2] Chief Design Engineer, McClelland Engineers, Inc., Houston, Tex.

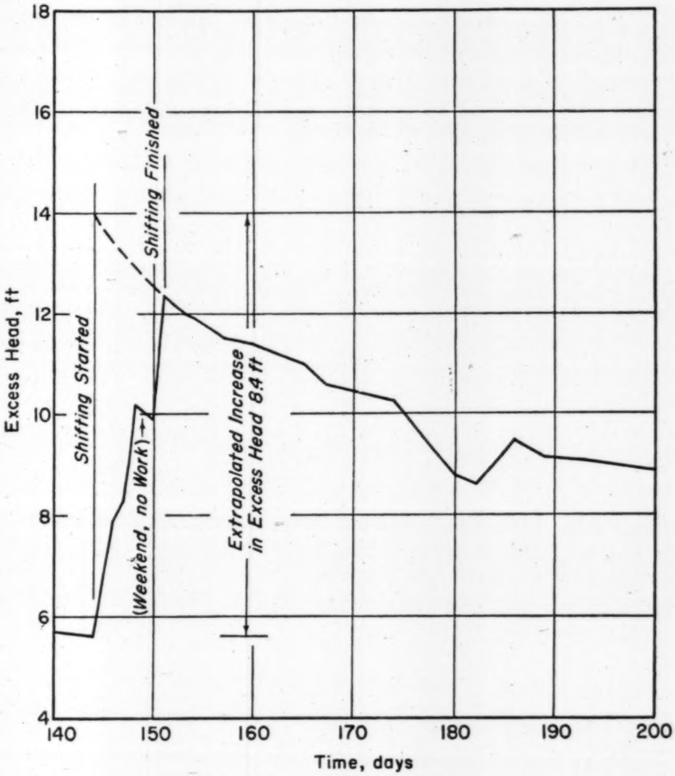


FIG. 10.—West Piezometric Level.

NOTE.—This is an expanded portion of Fig. 9 of the author's paper.

The difference between the shape of the actual time-settlement curve on a semilog plot and the S-shape curve based on Terzaghi's classical consolidation theory is evident to some degree on Fig. 8 of the author's paper. The theoretical curve shown on Fig. 8 was drawn to match the projected settlements of the the two "East" curves at the end of a 5-yr period. A more marked difference would be shown between the actual "West" curves for the initial 10 ft of fill and a corresponding theoretical curve. The time-settlement after fill was complete plots essentially as a straight line on the semilog graph, whereas theoretical time-settlement computed for a constant soil permeability plots in a very definite curve. Application of a theoretical solution for decreasing soil permeability might indicate a time-settlement relation more similar to that observed.

The author concludes from the changes

in pressure head as measured by the "West" piezometer when fill was shifted that the soil adjacent to the piezometer well was disturbed. Check tests were made at the time of piezometer installations which showed conclusively that the piezometers had no significant time lag. After an artificial change in the water level in a piezometer riser tube, the water level rapidly returned to its original position, confirming the author's conclusion that the in-place soil is much more permeable than is indicated by laboratory tests but not indicating any significant zone of low-permeability disturbed soil around the piezometer tip.

Replotting a portion of Fig. 9 of the author's paper to a larger scale on the accompanying Fig. 10 permits a closer examination of the changes of excess head shown by the "West" piezometer during and immediately after shifting of the fill. It should be borne in mind that the placement of 5 additional ft of fill required a period of 9 days. By reverse extrapolation of the piezometer curve after fill shifting was complete, as indicated by the dash line on Fig. 10, an indication is obtained that the "West" piezometer would have indicated a total excess head of 14 ft if the fill shifting had been done instantaneously. Thus, for instantaneous load application, the change in excess head would have been 8.4 ft, which corresponds very closely in terms of lb per sq ft to the 5 ft of added fill. Therefore, it is concluded that the piezometers were indicating the pore pressure in the clay at the piezometer tip with very little, if any, inherent error.

Comparison of the excess head measured by the piezometers at 25 days on the author's Fig. 9 with the applied load shows that the primary consolidation at the piezometer tips ranged from 30 to 67 per cent, with an average for the six piezometers of 47 per cent. Since the piezometers were at the center of the compressible stratum, these figures correspond to an average primary consolidation throughout the entire compressible stratum of 81 to 97 per cent (averaging 90 per cent). At this same time the average degree of consolidation computed from observations on the settlement plate adjacent to the six piezometers ranged from 24 to 48 per cent with an average of 33 per cent. Thus, only 10 days after fill placement was complete, the piezometers indicated that primary consolidation was practically complete even though the actual settlement was only one third of ultimate (and even less than half of the total settlement which was achieved in the limited preload period). A similar analysis can be made of the data from the "West" piezometer after fill shifting. The excess head measured at the center of the stratum at the end of fill shifting corresponds to an average primary consolidation of 70 per cent under the additional load; the degree of consolidation under the extra 5 ft of fill at this time as measured by a settlement plate was less than 25 per cent.

From the foregoing discussion and the data presented in this paper, it is concluded that the substantial difference between observed and computed time-settlement curves is not due exclusively to sample disturbance causing low laboratory consolidation rates. An additional probable cause may be that the conventional primary consolidation theory is not appropriate for the soils at the sites studied to convert time-consolidation for thin laboratory samples to time-settlement in the field.

MR. E. J. ZEGARRA (*author's closure.*)—Messrs. Reynolds and Focht are, in a way, participants in the preparation of the paper because of their association in the studies and investigations of the projects. Their knowledge of properties and characteristics of the soils involved is the basis for their observations.

With respect to the Delaware Project,

Mr. Reynolds points to the close correlation between predicted and observed settlements if the latter are extrapolated to 6 months and observes, in explanation, that laboratory consolidation tests do contain secondary time effects which are incorporated in estimating time settlements. This, however, does not explain the divergence in magnitude for a 3-month period. Mr. Reynolds concedes that there is an inconsistency. The reason common to the entire field consolidation process must, therefore, lie elsewhere. As pointed out in the paper, the stratification and random anisotropy of soils in general are not taken into account in the assumptions (Terzaghi, 1943, "Theoretical Soil Mechanics") of the theory of consolidation, and since these factors are obscured in the laboratory tests the results are of necessity different in the field.

It would be highly improper, as Mr. Reynolds warns, to reduce the total estimated settlement by an arbitrary amount. This could only be done after more field evidence is available through publication of actual records where the discrepancies are significant, and after research and theoretical study are given to this problem.

Mr. Focht has taken the behavior of the "West" piezometer at the time when loads were transferred to illustrate his contention respecting the shape of the curve and magnitude of settlement in the field.

In the computations furnished by Mr. Focht, the basis of comparison is the predicted figure obtained from laboratory tests. Whereas the piezometer readings indicated that after 10 days of loading a high percentage (90 per cent) of consolidation had taken place, the settlement plates showed that only 30 per cent of the computed ultimate settlement had been achieved. The former is an absolute figure; the latter a comparative one. The hypothesis advanced in the paper reconciles these two widely separated ratios by suggesting that the 30 per cent settlement is, in fact, nearly the total settlement from primary consolidation. The divergence of results is strikingly illustrated in the shape and magnitude of settlement of the theoretical and observed time-settlement curves given in Figs. 2, 4, and 9 of the paper.

Mr. Focht's conclusions are basically in agreement with those given in the paper. The differences are only of degree: where Mr. Focht says that disturbances are not the exclusive cause of difference, the author says they are the principal reason. If disturbances in the laboratory were eliminated and if sufficient samples were tested, the theoretical and field settlements would probably be much closer on the average. In its present state the theory would seem to be inappropriate.

THE COMPRESSIVE STRENGTH OF REMOLDED NIAGARA CLAY*

By D. J. Bazett[1] and S. W. Smotrych[1]

Synopsis

Triaxial compression tests on drained and undrained samples were carried out on remolded Niagara clay. Samples were prepared both in the normally consolidated and overconsolidated states from material with an initial water content equal to the liquid limit. Strain rate was investigated, and the effects of the membrane and drains and bushing friction were taken into account.

The results of the tests are reported in the form of consolidation data, stress paths, the variation of pore-pressure coefficient at failure with overconsolidation ratio, and compressive strength at failure with water content at failure.

Tests of drained and undrained samples were found to have common failure envelopes when the samples were normally consolidated or when the samples had the same maximum consolidation pressure.

In connection with research studies of the shearing strength of clay and the behavior of pore pressures, detailed data on normally consolidated and overconsolidated samples of remolded clay in drained and undrained triaxial compression tests have been obtained. The approach to this testing was influenced initially by discussions with D. J. Henkel at the Toronto Shear Strength Seminar (1958).

Because of limitations imposed by both time and the availability of testing facilities, it was necessary to make the tests on a single clay. Since the Hydro-Electric Power Commission of Ontario has had considerable practical experience with Niagara clay, it was selected for the study.

The results to date form a single unit in the study of saturated clays and will provide a control in the investigation of other variables.

Material

Niagara clay is a reddish-brown inorganic silty lacustrine clay, probably a deposit of glacial Lake Warren on the Niagara escarpment near Niagara Falls. The properties of the clay are as follows:

Liquid limit	41
Plastic limit	20
Specific gravity	2.76
Per cent finer than 2 μ	55

Material Preparation

In determining the condition of samples to be tested, it was decided that, in order to obtain normally consolidated samples, the samples should be consolidated from clay with a water content equal to or greater than the liquid limit and, to obtain a common consolidation line, that the samples should all be consolidated from identical initial moisture contents (**1**,**2**).[2]

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Supervising Engineer and Engineer, respectively, Soils Section, Structural Research Department, The Hydro-Electric Power Commission of Ontario (Canada).

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The method used to obtain samples with the desired water content was as follows: a bulk sample of the clay was mixed with distilled water, vibrated, and subjected to vacuum to remove air. A Harvard liquid limit device was used to control the water content at the liquid limit.

Mounting of Samples

The triaxial cells were fitted with two lines connecting to the sample pedestal, and these were initially deaired. A thin (0.006-in.) rubber latex membrane was sealed to the pedestal and held in position with a split brass former. Four $\frac{5}{16}$-in. filter paper drains[3] spaced equidistantly within the form were placed against the membrane and connected with the base stone to provide axial drainage. The clay was added in small units. Each unit was agitated, using thin rods, to cause the material to flow from the center of the mold to the outside and thus prevent the inclusion of air.

The nominal sample dimensions were a diameter of 2 in. and a height of 4 in. Once the sample had been brought to the desired height, the top cap was placed on the sample and the membrane was sealed to it. A negative head of 1.0 psi to induce a small negative pore pressure was applied to the sample for about 10 min by means of one of the lines to the pedestal. The valve was then closed and the split former removed. The sample height was measured, the cell was assembled, water was added to the cell, and the piston[4] was brought into contact with the sample. The cell pressure[5] was applied, the quantity of water expelled was measured in a graduated buret, and the change in height of the sample was gaged from the measured travel of the piston.

Normally consolidated samples were obtained by consolidating at cell pressures of 5, 10, 20, 40 and 80 psi. Consolidation time was 3 days.

Overconsolidated samples were obtained by reducing the cell pressures on samples to 40, 20, 10, and 5 psi from the common maximum consolidation pressure of 80 psi. The samples were allowed to expand for 2 days under the reduced pressures.

Three other overconsolidated samples were obtained with a maximum consolidation pressure of 100, 60, and 40 psi and reduced pressures of 25, 15, and 10 psi, respectively.

In earlier testing, two series of samples had been prepared, first by thumb-packing individual samples and then by driving tubes into consolidated material with a moisture content at which the samples could be conveniently handled.

It was found that the samples prepared by thumb-packing were not fully saturated, and both forms of samples behaved to some extent as overconsolidated material.

Undrained Test

The pore-pressure measuring system[6] was connected to one of the drainage lines, and deaired water was drawn through the base and pore-pressure measuring device to ensure the removal of air. This process was continued until no air could be detected in the pore pressure measuring system. The sample, membrane, and pedestal system were then tested for air. The cell pressure was increased, and the pore pressure was measured. A pore-pressure increase corresponding to the cell-pressure increase was obtained indicating that the whole system was saturated.

---

[3] Membrane and drain corrections were made to the measured stress (**3**). These ranged from 0.67 psi at 0.2-in. strain to 1.0 psi at 1.0-in. strain.

[4] Frictional losses through the bushing were measured and found to be in the order of 3 per cent of the applied load. This value has since been nominally applied.

[5] The cell pressure was controlled by use of a self-compensating mercury manometer (**3**).

[6] The pore-pressure measuring system used was similar to the system designed by the Norwegian Geotechnical Inst.

To determine a satisfactory strain rate for application in the undrained tests, three samples were consolidated at 80 psi and tested at different strain rates, namely, 25 per cent strain over $\frac{1}{2}$ hr, 2 hr, and 24 hr, respectively (Fig. 1). The difference between the results of the samples strained over 2 hr and over 24 hr was thought not to be significant, and it was decided, for convenience, to run the undrained tests at a strain rate of 25 per cent strain over $2\frac{1}{2}$ to 3 hr.

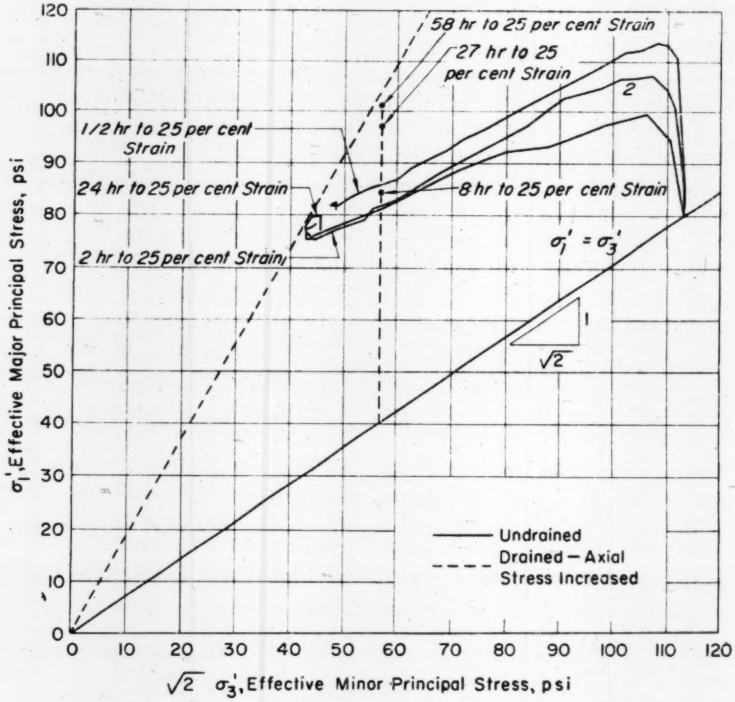


FIG. 1.—Stress Paths for Samples Tested at Varying Strain Rates.

DRAINED TEST

Drained tests were carried out using stress control (dead loads). The load increments were chosen on the basis of the undrained test results. These increments were limited by the possible pore pressures that would develop because of the loads. After loading, the sample was allowed to come to equilibrium before the succeeding load increment was applied, normally after 24 hr. Time to failure varied from about one to three weeks. Volume changes accompanying the tests were measured by means of a buret attached to the drainage line.

Three samples were each consolidated at 40 psi and tested to indicate the effect of strain rate. The strain rates were 25 per cent strain over 8, 27, and 58 hr. The results are shown in Fig. 1.

TEST RESULTS

Water content change calculated from volume change measured with the buret was not consistent with the water content change from direct measurements. These discrepancies were attributed to evaporation from the burets and leakage through and past the membrane. The water contents shown in Figs. 2 and 7 are from direct determination.

The results of tests of low compressive strength were somewhat erratic. This is attributed to inaccuracies in the low ranges of the measuring devices, and inaccuracies in membrane, drain and bushing corrections which become significant at low stresses.

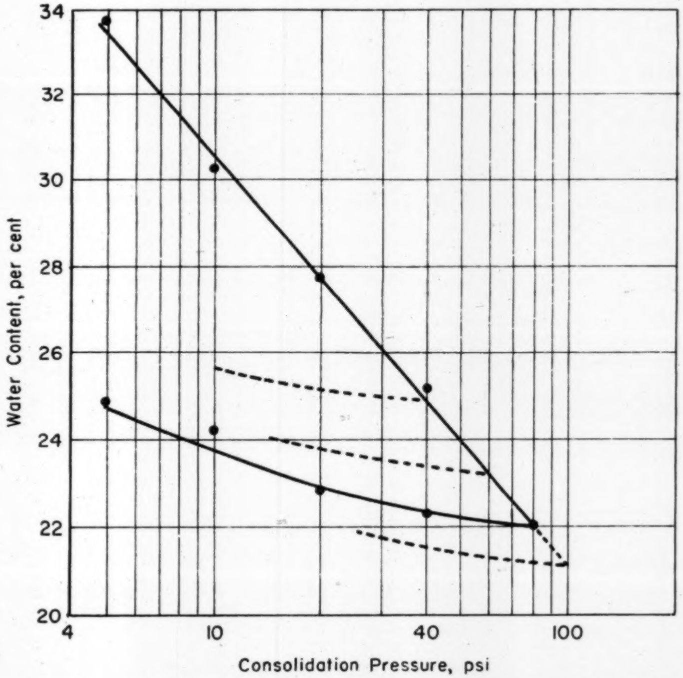


FIG. 2.—Relation Between Equilibrium Water Content and Consolidation Pressure.

Equilibrium water content is plotted against the logarithm of consolidation pressure in Fig. 2. Approximate rebound curves from pressures 100, 60, and 40 psi are included.

Shear planes were observed in most of the samples, both drained and undrained, and occurred at about 60 deg to the horizontal as measured at the completion of testing. Shear planes were generally not well defined and described as "nascent" in the normally consolidated specimen but were well developed and described as "mature" in the overconsolidated samples. Bulging occurred uniformly with the axial deformation to the end of the test except in those cases where a well-developed shearing surface was observed.

It has been found convenient and illustrative to plot the test results in stress space, Figs. 1, 3, 4, and 5 as recently reported by Henkel (4).

These graphs are obtained by plotting the values of effective major principal stress, (σ_1'), as ordinates, against the values of effective minor principal stress, (σ_3'), as abscissae. The σ_3' scale is distorted by a factor of $\sqrt{2}$. The line representing an equal all-round effective

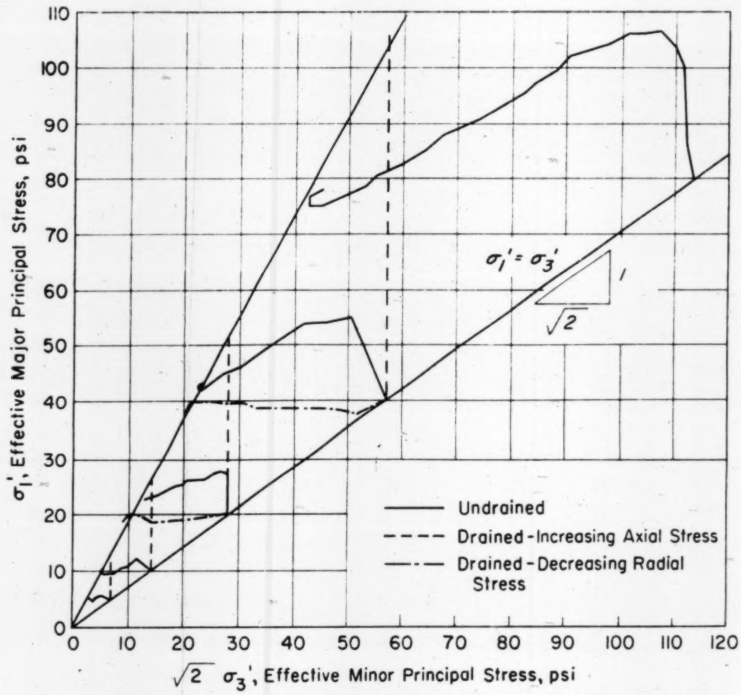


FIG. 3.—Stress Paths for Normally Consolidated Samples.

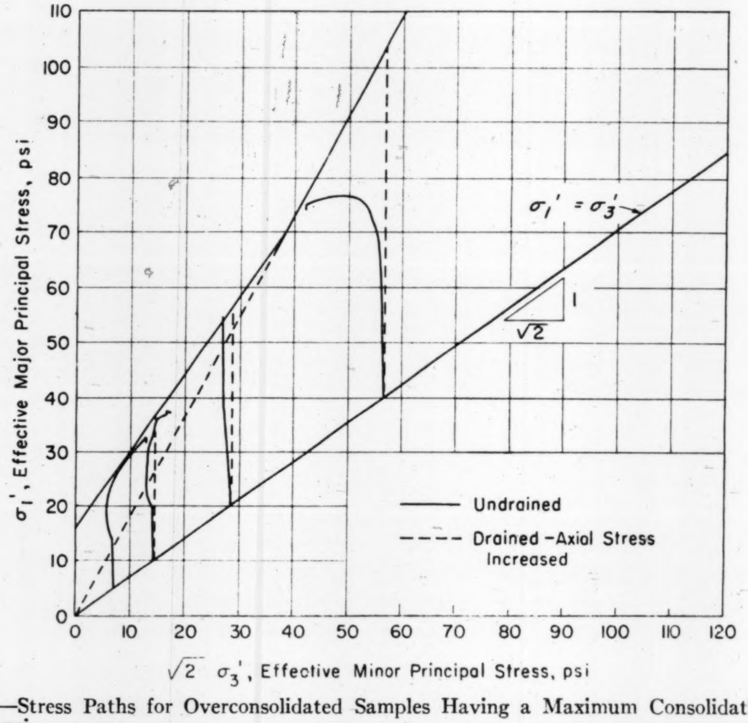


FIG. 4.—Stress Paths for Overconsolidated Samples Having a Maximum Consolidation Pressure of 80 psi.

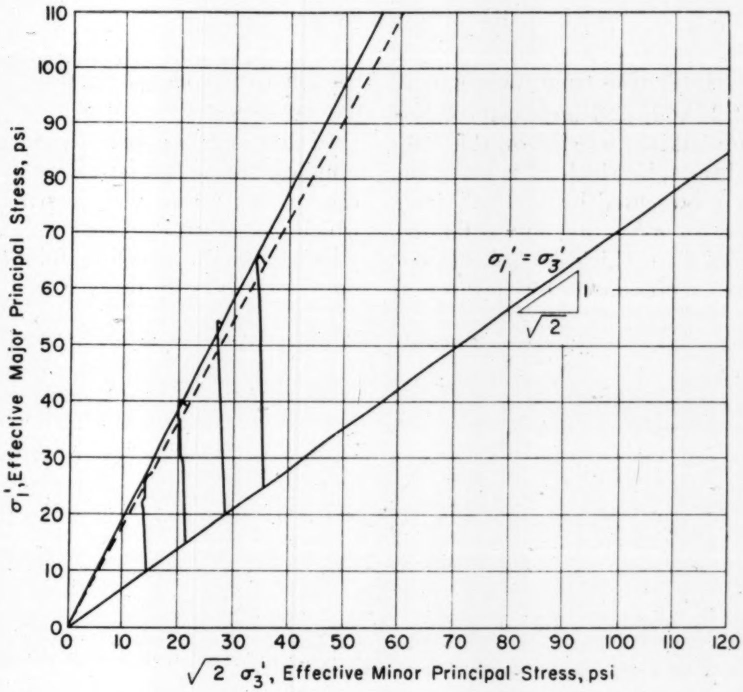


FIG. 5.—Stress Paths for Overconsolidated Samples Having an Overconsolidation Ratio of 4.

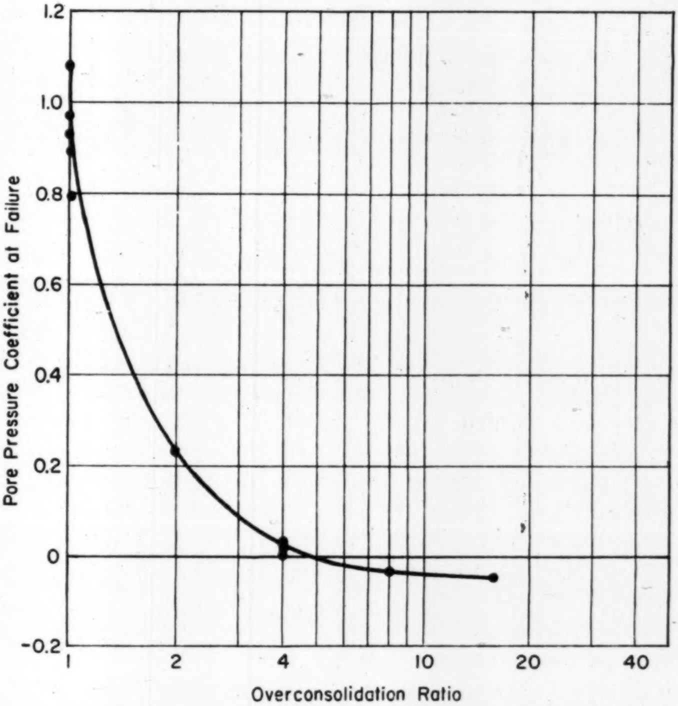


FIG. 6.—Variation in A_f with Overconsolidation Ratio.

stress ($\sigma_1' = \sigma_3'$) rises from the origin at a slope of $1/\sqrt{2}$, and any state of isotropic consolidation will fall on this line.

Drained tests, in which σ_3' is held constant, are represented by vertical stress paths from the $\sigma_1' = \sigma_3'$ line to failure. Drained tests, in which σ_1' is held constant, are represented by horizontal stress paths from the $\sigma_1' = \sigma_3'$ line to failure.

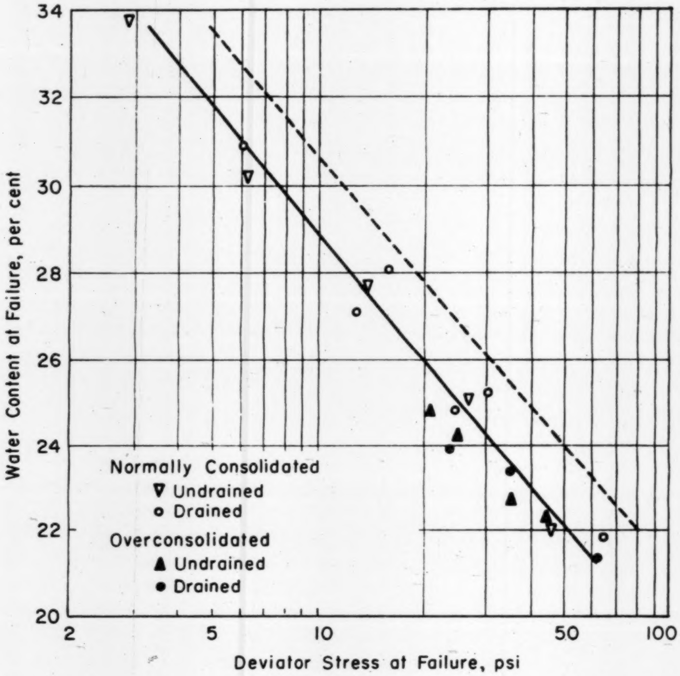


FIG. 7.—Relation Between Water Content and Compressive Strength At Failure $(\sigma_1 - \sigma_3)_f$.

Undrained tests, in which σ_1' and σ_3' both vary, are represented by curved paths from the $\sigma_1' = \sigma_3'$ line to the completion of the test.

An envelope similar to that drawn in the Mohr diagram can be used to relate a number of individual test results.

The slope of the envelope is a function of the angle of shearing resistance in terms of effective stresses, ϕ', and the intercept on the σ_1' axis is a function of the apparent cohesion in terms of effective stresses, c'.

In the testing of normally consolidated samples, the failure envelope found for the drained tests was a straight line which passed through the origin and was identical to the envelope found for the undrained tests. In terms of the effective shearing parameters, $\phi' = 26\frac{1}{2}$-deg (Fig. 3.)

The failure envelopes for the stress paths of the overconsolidated samples with the same maximum consolidation pressure were found to be identical both for the drained and the undrained tests and did not pass through the origin (Fig. 4).

The failure envelope of a set of samples overconsolidated at the same ratio of consolidation pressure to rebound pressure (overconsolidation ratio) was also found to be a straight line passing through the origin (Fig. 4(c)). The shapes of the stress paths were similar as were the values of pore-pressure coefficient A_f (the ratio of pore pressure to deviator stress at failure (5)).

The pore-pressure coefficient at failure, A_f, is shown plotted against the overconsolidation ratio in Fig. 6. It may be seen that A_f decreases with increasing overconsolidation ratio (6).

The logarithm of deviator stress with water content at failure is shown in Fig. 7 for normally-consolidated and overconsolidated samples tested in drained and undrained tests. The values for normally consolidated samples tested in drained and undrained tests approximate a straight line parallel to the consolidation line (7). Although the values for overconsolidated samples tested in drained or undrained tests approximated the same straight line as found for the normally consolidated samples (8), the use of such a line can be shown to imply a value of Hvorslev's parameter ϕ_r equal to zero and this is not consistent with the measured angles of the shear planes.

CONCLUSIONS

Drained and undrained tests had common failure envelopes when the samples were normally consolidated or when the samples had the same maximum consolidation pressure.

REFERENCES

(1) L. Bjerrum, "Theoretical and Experimental Investigations on the Shear Strength of Soils," Norwegian Geotechnical Inst., Oslo, Publication No. 5 (1954).

(2) A. W. Skempton, "Notes on the Compressibility of Clays," *Quarterly Journal*, Geological Society of London (1944).

(3) A. W. Bishop and D. J. Henkel, "The Measurement of Soil Properties in the Triaxial Test," Edward Arnold, Ltd. (1957).

(4) D. J. Henkel, "The Relationships Between the Strength, Pore-Water Pressure and Volume-Change Characteristics of Saturated Clays," *Geotechnique*, Vol. 9, pp. 119–135 (1959).

(5) A. W. Skempton, "The Pore Pressure Coefficients A and B," *Geotechnique*, Vol. 4, pp. 143–152 (1954).

(6) D. J. Henkel, "Overconsolidation on Clays During Shear," *Geotechnique*, Vol. 6, pp. 139–150 (1956).

(7) P. C. Rutledge, "Cooperative Triaxial Shear Research Program of the Corps of Engineers," Waterways Experiment Station (1947).

(8) D. W. Taylor, "Fundamentals of Soil Mechanics," John Wiley & Sons, Inc., New York, N. Y. (1948).

DISCUSSION

Mr. W. E. Schmid.[1]—Were the pore pressures measured in order to get the effective stresses measured at the base through the porous stone or somewhere in the sample?

When the authors state that the change in volume as observed from a buret did not correspond to the change in volume by direct measurement, how was the direct measurement made?

Mr. S. W. Smotrych (*author*).—With regard to the first question, we made our pore pressure measurements at the base through a porous stone. The pore pressures measured this way for a sample tested at a strain rate of 2 hr to 25 per cent strain were found similar to those for another sample tested at a strain rate of 24 hr to 25 per cent strain as shown in Fig. 1 of the paper. It was taken that at the strain rate of 2 hr to 25 per cent strain the pore pressures were essentially equalized throughout the sample. It was decided, consequently, to perform the undrained tests at a strain rate of 2½ to 3 hr to 25 per cent strain.

In answer to the second question, water content determinations were made on portions of the samples before and after consolidation and testing. The water content changes determined in this way were referred to as being made on direct determinations. Water content changes using the volume change measured in the buret did not check with those by direct determinations, particularly in the drained tests.

Mr. Schmid.—Did the volume change fail to correlate during consolidation?

Mr. Smotrych.—The consolidation behavior was determined from the water content determinations done for undrained tests. The correlation of water content change determined from volume change measured in the buret and by direct determination was better in the undrained tests. This is thought due to the shorter time involved in the undrained tests, thus any leakage through or by the membrane or evaporation from the buret would be less pronounced.

Mr. T. Cameron Kenney[2] (*by letter*).—The authors have presented the results of a comprehensive triaxial testing program on a soil from southwestern Ontario, the origin of which has been suggested to be lacustrine. There are many locations in this part of Canada where deposits of soft, sometimes normally consolidated, soils exist to depths greater than 100 ft. A peculiar feature of some of these deposits is their extreme homogeneity on a macroscopic scale whereas on a microscopic scale they are found to consist of a wide range of particle sizes from gravel to clay. On the basis of their naturally soft condition, some people class these soils as lacustrine deposits; on the basis of their particle size distribution other people class them as tills. It would be of considerable interest if the authors would present the particle size distribution curve for natural Niagara Clay and also give the reasons why they believe it to be probably a lacustrine clay.

---

[1] Associate Professor of Civil Engineering, Princeton University, Princeton, N. J.

[2] Engineer, H. G. Acres & Co., Ltd., Niagara Falls, Canada.

The compressive strength of a soil at failure, $(\sigma_1 - \sigma_3)_f$, can be expressed in terms of true cohesion, C_r, and true angle of internal friction, ϕ_r, in the following manner:

$$(\sigma_1 - \sigma_3)_f = 2C_r\left(\frac{\cos\phi_r}{1-\sin\phi_r}\right) + 2\sigma_3{}'_f\left(\frac{\sin\phi_r}{1-\sin\phi_r}\right) = 2\chi\cdot\rho_e\left(\frac{\cos\phi_r}{1-\sin\phi_r}\right) + 2\sigma_3{}'_f\left(\frac{\sin\phi_r}{1-\sin\phi_r}\right)\ldots(1)$$

or

$$\left(\frac{\sigma_1{}'}{\sigma_3{}'}\right)_f = 2\chi\left(\frac{\rho_e}{\sigma_3{}'_f}\right)\left(\frac{\cos\phi_r}{1-\sin\phi_r}\right) + \left(\frac{2\sin\phi_r}{1-\sin\phi_r}\right) + 1\ldots(2)$$

where:

ρ_e = the equivalent normal consolidation pressure corresponding to the water content of the sample at failure, and

χ = a constant relating normal consolidation pressure and true cohesion.

When analyzing their data, the authors found that the compressive stress at failure seemed to be independent of the stress history of the sample and, therefore, was independent of ρ_e. Their conclusion was that this result implies that $\phi_r = 0$, whereas from Eq 1 it is seen that the implication is that $C_r = 0$. Actually, neither of these implications is correct because the compressive stress at failure was not independent of the stress history of the sample, and this is clearly shown in Figs. 4 and 5 of the paper where overconsolidated samples were found to exhibit higher shear strengths than normally consolidated samples. As shown in Figs. 3 and 5, soils having the same overconsolidation ratios also have constant values of $\left(\frac{\sigma_1{}'}{\sigma_3{}'}\right)_f$ and $\frac{\rho_e}{\sigma_3{}'_f}$. From these figures, Eq 2 has been evaluated for the cases of overconsolidation ratios of unity and 4.0 as follows:

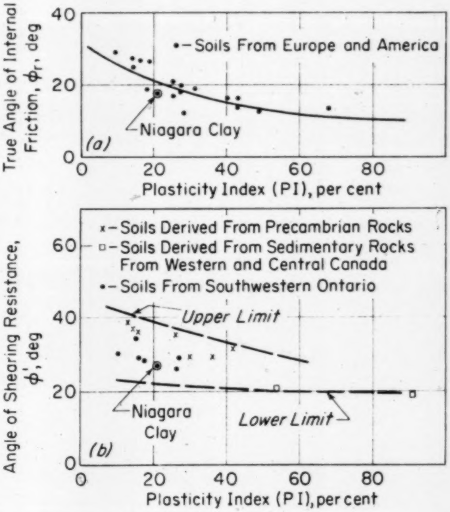


FIG. 8.—(*a*) Relationship Between ϕ_r and Plasticity Index for Remolded Soils. (*b*) Relationship Between ϕ' and Plasticity Index for Remolded Normally-Consolidated soils.

(*a*) *Overconsolidation ratio* = *1.0* (*Fig. 3*):

$$2.57 = 2\chi(2.71)\left(\frac{\cos\phi_r}{1-\sin\phi_r}\right) + \left(\frac{2\sin\phi_r}{1-\sin\phi_r}\right) + 1$$

(*b*) *Overconsolidation ratio* = *4.0* (*Fig. 5*):

$$3.28 = 2\chi(3.46)\left(\frac{\cos\phi_r}{1-\sin\phi_r}\right) + \left(\frac{2\sin\phi_r}{1-\sin\phi_r}\right) + 1$$

Assuming that ϕ_r remains constant, these expressions were solved simultaneously and the following results were obtained: $\phi_r = 17.5$ deg, and $\chi = 0.10$.

The value of $\phi_r = 17.5$ for a clay having a plasticity index of 21 per cent seems to be consistent with the results of other published data summarized in the accompanying Fig. 8.

Also in Fig. 8 are summarized the angle of shearing resistance, ϕ', of some Canadian soils and the upper and lower limits of this parameter obtained from a review of published data. The Canadian soils seem to extend over the entire range between the limits. It is interesting to make the following observations:

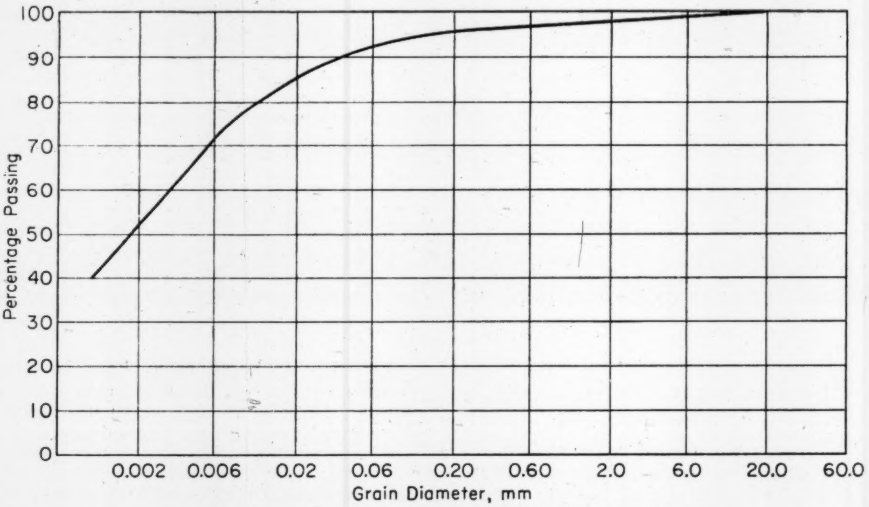


FIG. 9.—Mechanical Analysis of Natural Niagara Clay.

1. The soils primarily derived from Precambrian rocks exhibit high values of ϕ', whereas those soils derived from the soft sedimentary rocks of central and western Canada exhibit low values of ϕ'.

2. The soils found in southwestern Ontario, which are probably primarily derived from the local sedimentary rocks, generally exhibit ϕ' values between the two groups mentioned above. The point $\phi' = 26.5$ deg, plasticity index = 21 per cent for Niagara Clay, seems to fit in well with the other data.

The authors are to be thanked for presenting such a complete record of triaxial testing on this Canadian clay.

MESSRS. D. J. BAZETT AND S. W. SMOTRYCH (*authors' closure*).—The material used in the study was a reddish brown inorganic silty clay with little or no evidence of sand or gravel. A grading curve is shown in Fig. 9. The deposit, from a quarry about 5 miles west of Niagara Falls, was 10 to 15 ft thick. It was located on stratified bedrock and covered with only a thin layer of humus No varving was visible and the clay appeared to be homogeneous.

The location from which the material was taken is close to the boundary of a moraine as mapped by Chapman and Putnam.[3] It is typical of the reddish brown silty clays found extensively in the Niagara area and indicated in their mapping as lacustrine. No detailed study of the geological aspect of the clays was made but it has been supposed that they were deposited during the recession of the Wisconsin Glacier at a higher elevation than and prior to the Lake Iroquois stage. The deposits thus seem to be from Lake Whittlesey or, more probably, Lake Warren.

---

[3] L. J. Chapman and D. F. Putnam, "The Physiography of Southern Ontario," University of Toronto Press, Toronto, Canada, 1951.

To determine the values of C_r and ϕ_r, it is necessary to compare the results of tests in which the samples have the same water content at failure but different effective stresses. These conditions may be achieved in a series of tests on normally- and overconsolidated samples. With Niagara Clay, which has a low plasticity index, the separation between the compressive strengths for normally- and lightly overconsolidated samples at the same failure water content is small.

If the separation is zero, it would indicate a value of $\phi_r = 0$. The authors did not wish to imply that they believed this to be the case and their determination of ϕ_r is discussed below.

Mr. Kenney has shown an interesting implication of the authors' experimental observation that "the failure envelope of a set of samples overconsolidated at the same overconsolidation ratio was a straight line passing through the origin" and uses this straight-line relationship (constant σ_1'/σ_3') to determine the Hvorslev parameters.

If the straight-line relationship noted by the authors is general, Mr. Kenney's method offers a very neat method of determining values of ϕ_r and χ from a limited number of tests.

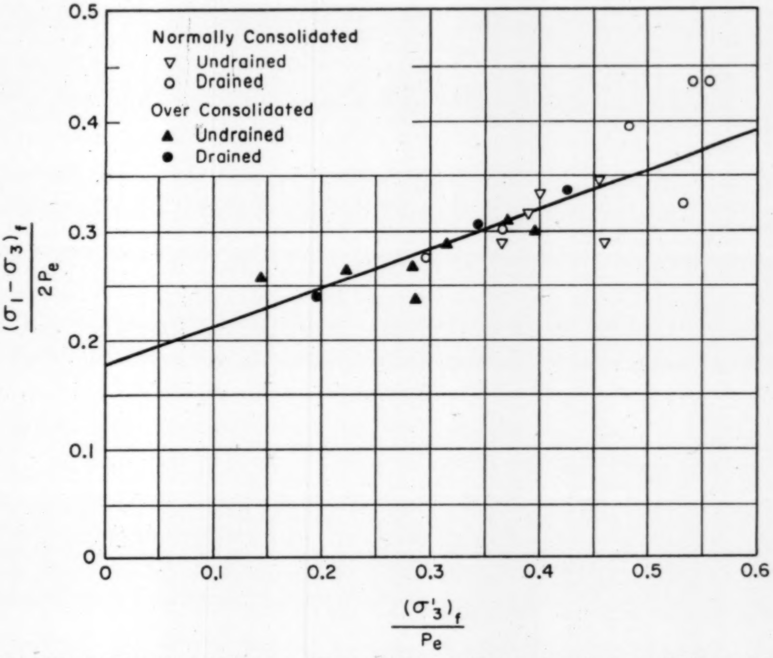


FIG. 10.—Plot of Results of Tests on Niagara Clay to Determine Average Values of the True Angle of Internal Friction and of the Ratio of True Cohesion to Equivalent Consolidation Pressure.

The authors have calculated the values of ϕ_r and χ following the method suggested by Bishop and Henkel[4] which has the advantage of using all the available test results. The basic shear strength equation is rewritten in terms of the principal effective stresses as follows:

---

[4] A. W. Bishop and D. J. Henkel, "The Measurement of Soil Properties in the Triaxial Test," Edward Arnold, Ltd., (1957).

$$\frac{(\sigma_1 - \sigma_3)_f}{2\rho_e} = \frac{C_r \cos \phi_r + \sigma_3{}'_f \sin \phi_r}{\rho_e(1 - \sin \phi_r)}$$

This equation permits a dimensionless plot of all the test results as shown in Fig. 10 so that satisfactory average values of the parameters may be obtained. Solving for the parameters from the results of the plot, $\phi_r = 20$ deg and $\chi = 0.12$.

The authors are thankful for Mr. Schmid's and Mr. Kenney's pertinent questions and comments.

DUPLICATION OF PROTOTYPE STRESS-STRAIN RELATIONS IN SOIL MASSES BY LABORATORY TESTS*

By Richard G. Ahlvin[1] and Donald N. Brown[2]

Synopsis

Studies of the distribution of stress and strain in soil masses, conducted by the U. S. Army Engineer Waterways Experiment Station (WES), have included two full-scale test sections. Loads—of the magnitude of current aircraft wheel loads—were applied to these test sections, and the stresses and deflections were measured. From these, stress-strain curves have been developed in two cases which are representative of prototype conditions.

Any rational analytical method involving soil stress-strain relations will require a means of evaluating the stress-strain characteristics of soil from laboratory specimens or simple tests. For this reason, attempts were made to develop test procedures in the laboratory which would yield stress-strain curves substantially the same as those from the two test sections.

This paper presents and points out the importance of the two prototype stress-strain relations and describes some of the laboratory test methods used in attempting to duplicate the prototype curves. Methods are presented by which each of the prototype stress-strain curves were substantially duplicated in laboratory tests.

The tests described in this paper were conducted by the Waterways Experiment Station as a part of the Corps of Engineers comprehensive study of the distribution of stress, strain, and deflection produced in a large homogeneous soil mass by application of loads to the surface of the mass. One objective of the comprehensive study was to develop a method of performing tests on small soil samples in the laboratory whereby the actual stress-strain relations for a large soil mass could be predicted. The specific purpose of the triaxial test program described herein was to establish a laboratory test that would produce stress-strain curves duplicating stress-strain curves developed from pressure-cell and deflection-gage measurements within a homogeneous subgrade constructed with conventional field equipment.

In studies conducted in the investigation of pressures and deflections under flexible pavements, two field stress-strain curves were developed for homogeneous soil masses subjected to surface loading. One curve (curve 1, Fig. 1) was developed from stresses and deflections measured at points within a large, homogeneous clayey-silt field test section and is, therefore, representative of the actual stress-strain relations existing within that soil mass. The other curve (curve 2, Fig. 1) was developed from stresses and deflections measured at points within a large, air-dried, homogeneous sand field test section and is, therefore, representative of the actual stress-strain relations existing within that soil mass. These two field stress-strain curves provided field data, heretofore unavailable, with which stress-strain curves developed in laboratory tests could be compared.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Chief, Special Projects Section, Soils Division, U. S. Army Engineer Waterways Experiment Station, Vicksburg, Miss.

[2] Engineer, Special Projects Section, Soils Division, U. S. Army Engineer Waterways Experiment Station, Vicksburg, Miss.

The methods and procedures used in constructing the full-scale test sections and conducting the tests from which these field stress-strain curves were developed are described only briefly in this paper. A detailed description is given in two Waterways Experiment Station publications.[3,4,5]

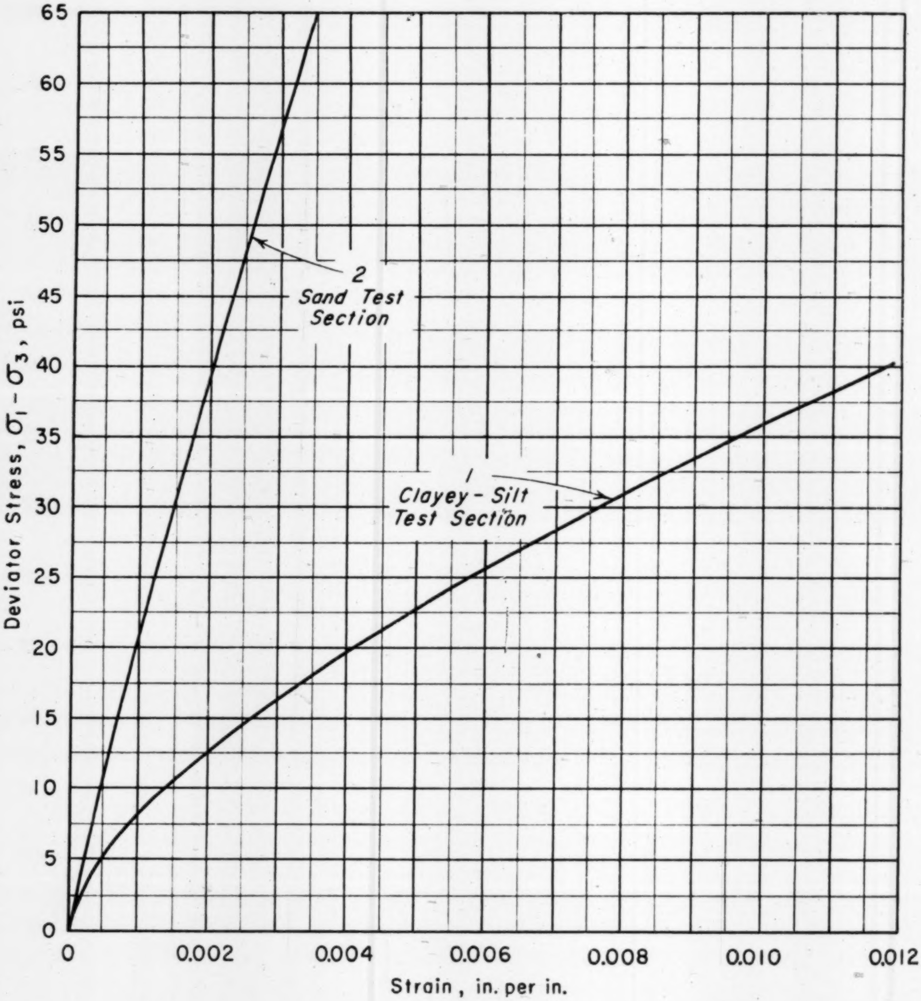


FIG. 1.—Field Stress-Strain Curves Developed for Two Homogeneous Soil Masses Subjected to Surface Loading.

---

[3] "Investigations of Pressures and Deflections for Flexible Pavements," Report No. 1: "Homogeneous Clayey-Silt Test Section," March 1951; Report No. 4: "Homogeneous Sand Test Section," Dec. 1954, *Technical Memorandum No. 3-323*, Waterways Experiment Station, Vicksburg, Miss.

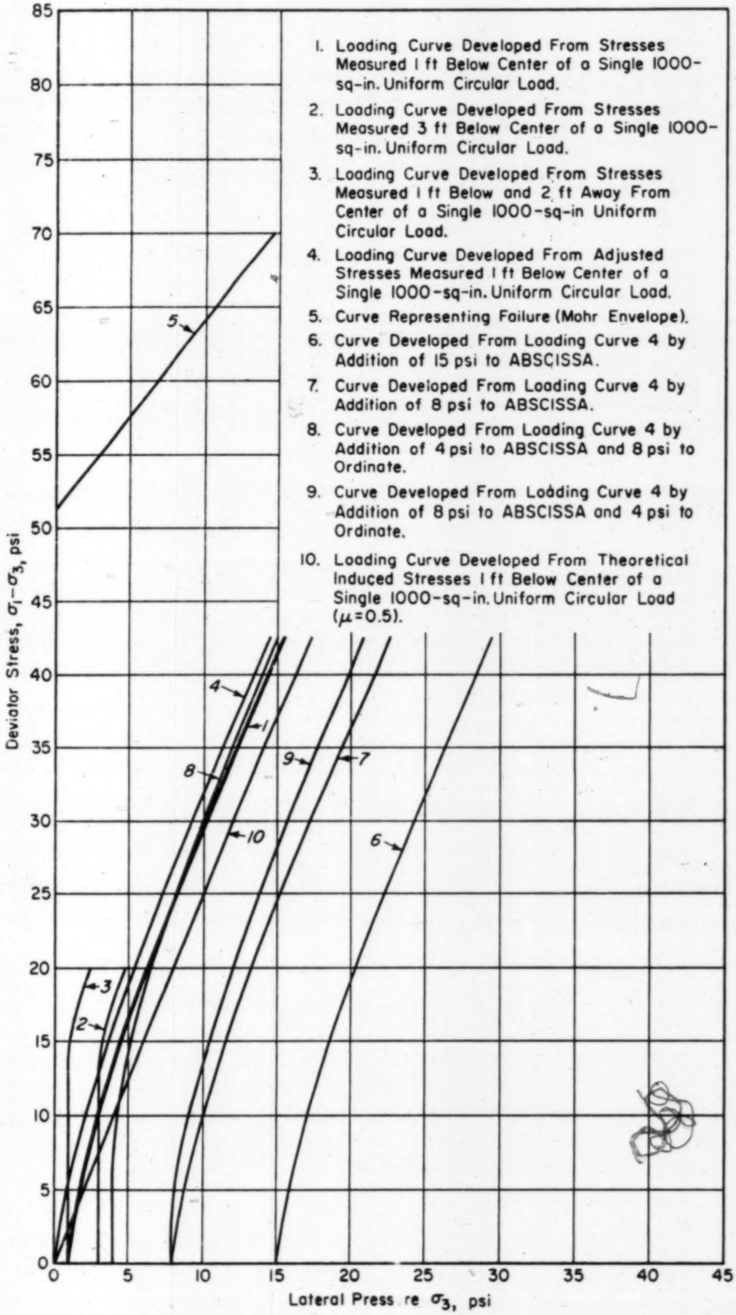


FIG. 2.—Loading Curves Developed in Attempts to Duplicate Homogeneous Clayey-Silt Test Section Stress-Strain Curve.

The first full-scale field test section was constructed with conventional construction equipment to represent a typical cohesive subgrade having a California Bearing Ratio (CBR) value of about 10. The material used in this section was a clayey-silt soil, classified CL according to the Unified Soil Classification System, having an average liquid limit of 36 and a plasticity index of 12.

The second test section was constructed with conventional construction equipment to represent a typical cohesionless subgrade compacted to a relative density of about 85 per cent. The material used in this section was a fairly uniform stream-deposited fine-to-medium sand (SP).

Uniform circular loads were applied to the surface of the homogeneous soil test sections by means of flexible-face, water-filled, circular plates that were developed especially for these studies. The flexible plates were made in three sizes, 250, 500, and 1000 sq in. Loads were applied to the flexible plates by means of hydraulic jacking equipment designed for application of loads through any combination of four hydraulic jacks connected to a central pumping mechanism. The reaction for the hydraulic jacks was a specially designed steel truss which spanned the test sections and provided load reaction up to 250,000 lb. Pressure cells and deflection gages were used to measure stresses and deflections in the test sections. Measurements were made at offsets up to 10 ft and depths up to 5 ft from the center of the loading plate and surface of the test section, respectively. At each point of measurement, stresses were determined in the direction of the principal axes, and vertical deflections were measured. Vertical deflections were plotted versus depth of reading and slopes of the resulting curves at each point of measurement determined the vertical strain at that point. This permitted plotting of deviator stress, $\sigma_z - \mu (\sigma_x + \sigma_y)$, versus vertical strain, ϵ_z, which yielded the field stress-strain relations shown in Fig. 1. These relations represent the actual stiffness or stress-strain relations effective in the prototype-scale test sections subject to full-scale loadings.

LABORATORY TESTS

Clayey-Silt Test Section:

Five series of the following triaxial-type tests were conducted on undisturbed samples from the clayey-silt test section:

(*a*) Quick (unconsolidated, undrained) triaxial test;
(*b*) Confined-compression test;
(*c*) Triaxial-cell test; and
(*d*) Variable-confining-pressure (VCP) triaxial test.

Comparison of stress-strain curves developed from quick triaxial, confined-compression, and triaxial-cell tests on undisturbed samples cut from the test section with the field stress-strain curve (curve 1, Fig. 1) showed that duplication of the field stress-strain curve had not been accomplished in these tests.

Before further laboratory tests were conducted, five basic loading curves were developed. Three of these curves were developed from stresses measured within the test section during application of the uniform, circular test loads. Vertical-lateral stress relations measured during loading were used to develop loading curves for points within the test section: (*a*) 1 ft below the center of a 1000-sq-in., uniform, circular load, (*b*) 3 ft below the center of a 1000-sq-in., uniform, circular load, and (*c*) 1 ft below and 2 ft away from the center of a 1000-sq-in., uniform, circular load (curves 1, 2, and 3

---

[4] C. R. Foster and S. M. Fergus, "Stress Distribution in a Homogeneous Soil," Highway Research Board *Research Report No. 12-F*, 1951.

[5] W. J. Turnbull, A. A. Maxwell, R. G. Ahlvin, "Stresses and Deflections in Homogeneous Soil Masses," prepared for presentation at 5th International Conference on Soil Mechanics and Foundation Engineering, Paris, July 1961.

of Fig. 2. respectively). The fourth curve (curve 4, Fig. 2) was developed from induced stresses (overburden and residual stresses not included) measured 1 ft below the center of a 1000-sq-in., uniform, circular load. This curve passes through the origin and is parallel to curve 1 of Fig. 2. The fifth of these basic loading curves (curve 5, Fig. 2) was developed from data obtained from six quick triaxial tests (first series of tests) performed on undisturbed samples from the test section. This curve represents failure conditions (Mohr envelope).

The fourth series of tests consisted of VCP quick triaxial tests performed on undisturbed samples from the test section. Prior to the tests, each sample was prestressed with one cycle of axial loading at 5 psi (seating load) and three cycles of stress equal to two thirds of the maximum stress difference (deviator stress) measured in the test section at the pertinent point during loading. The lateral pressure used to confine the sample during cyclic loading was constant for any one test and was based on the overburden stress measured in the test section in all cases except one in which a lateral pressure of 15 psi was used. After the sample was prestressed by cyclic loading, a VCP triaxial test was made. Loading curves 1, 2, and 6 of Fig. 2 were used as guides during loading of the samples in this series of tests. Stress-strain curves developed from the tests using loading curves 1 and 2 approached duplication of the field stress-strain curve, but the slope of both curves was somewhat flatter than the field stress-strain curve. The stress-strain curve developed from the test using loading curve 6 also approached duplication, but its slope was considerably steeper than the field stress-strain curve.

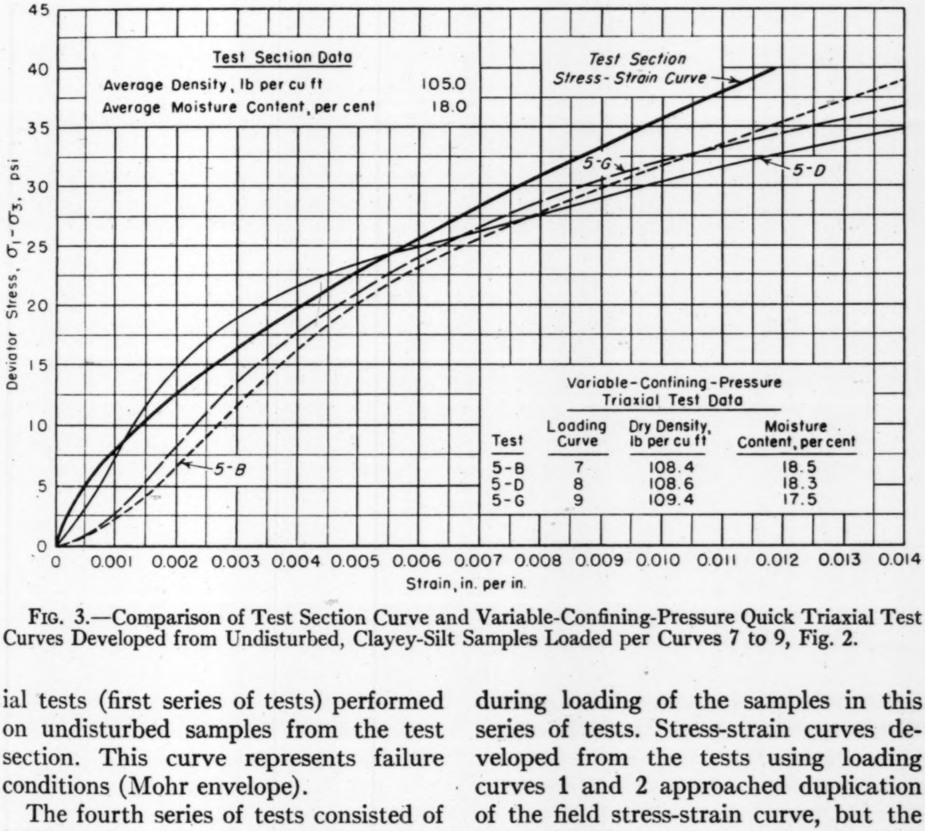


FIG. 3.—Comparison of Test Section Curve and Variable-Confining-Pressure Quick Triaxial Test Curves Developed from Undisturbed, Clayey-Silt Samples Loaded per Curves 7 to 9, Fig. 2.

Since the VCP quick triaxial tests that

were performed using loading curves 1 and 6 in Fig. 2 as guides resulted in stress-strain curves that bracketed the field test section stress-strain curve, it seemed reasonable to use a loading curve that would fall somewhere between curves 1 and 6 in a further attempt to duplicate the field stress-strain curve. Accordingly, the fifth series of tests consisted of VCP quick triaxial tests performed on undisturbed samples cut from the test section in which arbitrary increments of vertical and lateral stress were added to loading curve 4, Fig. 2. Three tests of this type were made for each of three loading conditions as shown by loading curves 7, 8, and 9, Fig. 2. Figure 3 shows three stress-strain curves resulting from tests 5-*B*, 5-*D*, and 5-*G*, which are typical of the stress-strain curves developed from this series of tests, compared with the test section stress-strain curve. Loading curves 7, 8, and 9 were used as loading guides during performance of tests 5-*B*, 5-*D*, and 5-*G*, respectively. Though not exactly duplicating the field stress-strain curve, curves 5-*B* and 5-*G* do approach duplication so closely that they may be considered, for practical purposes, to be the same.

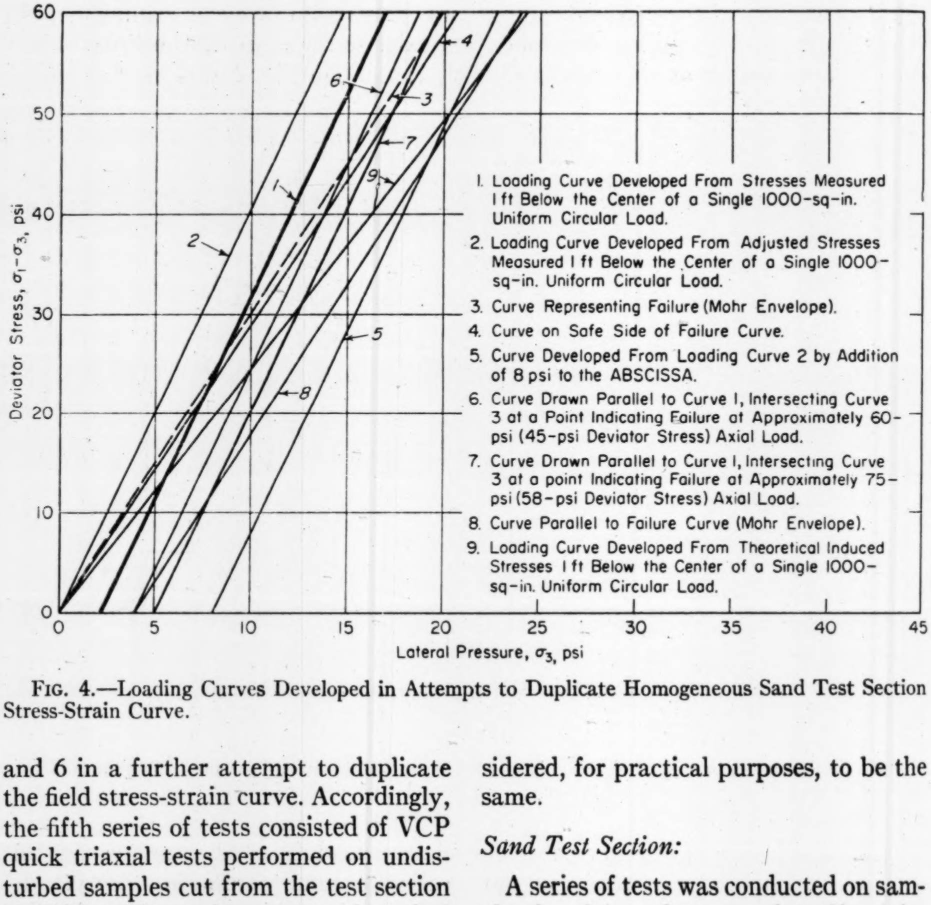


FIG. 4.—Loading Curves Developed in Attempts to Duplicate Homogeneous Sand Test Section Stress-Strain Curve.

Sand Test Section:

A series of tests was conducted on samples for the sand test section. Six series of the following triaxial-type tests were performed on remolded samples:

(*a*) Constant-confining-pressure (CCP) triaxial test;

(*b*) Variable-confining-pressure (VCP) triaxial test; and

(*c*) Variable-confining-pressure (VCP) triaxial test on prestressed sample.

The first series of tests consisted of nine CCP triaxial tests performed on remolded samples from the sand test section. The stress-strain curves developed from this series of tests did not duplicate the field stress-strain curve.

Prior to performing further laboratory tests, three basic loading curves were developed. The first of these curves was developed from stresses measured by pressure cells and deflection gages buried within the test section. Vertical-lateral stress relations measured within the test section were used to develop loading curves for points (*a*) 6 in. below the center of a 1000-sq-in., uniform, circular load, (*b*) 1 ft below the center of a 1000-sq-in., uniform, circular load, and (*c*) 3 ft below the center of a 1000-sq-in., uniform, circular load.

The loading curves developed for each of these points indicated substantially the same vertical-lateral stress relations; therefore, the loading curve developed from measured stresses and strains 1 ft below the center of a 1000-sq-in., uniform, circular load (Fig. 4, curve 1) was chosen as a loading guide in further testing. The second loading curve (curve 2) was developed from induced stresses (overburden and residual stresses not included) measured 1 ft below the center of a 1000-sq-in., uniform, circular load. This curve passes through the origin and is parallel to curve 1. The third loading curve (curve 3) was developed from data obtained from nine CCP triaxial tests (first series of tests) performed on remolded samples from the test section. This curve represents failure (Mohr envelope). A comparison of curve 1 with curve 3 indicates stress obliquities that are larger than are considered possible in the Mohr failure hypothesis. Reasons for this difference between measured and theoretical stress relations have been discussed in *Technical Memorandum No. 3-323*[3] and are not repeated in this paper.

The second series of tests consisted of three VCP triaxial tests performed on remolded samples of sand compacted to three different densities (111, 108.5, and 106 lb per cu ft). In each test the confining pressure was made to vary as indicated by curve 1, Fig. 4. None of the stress-strain curves developed from these tests approached duplication of the field stress-strain curve.

The third series of tests was identical with the second series except that curve 4, Fig. 4, was used to determine the variable confining pressure during performance of this series of tests. None of the stress-strain curves developed from these tests approached duplication of the field stress-strain curve.

The fourth series of tests consisted of three VCP triaxial tests performed on remolded samples in which the confining pressure during testing was varied as shown by curve 5, Fig. 4. This curve corresponds to the loading curve which gave the best results in tests on undisturbed samples from the clayey-silt test section. Stress-strain curves developed from this series of tests approached duplication of the field stress-strain curve, but still better agreement was desired.

The fifth series of tests consisted of six VCP triaxial tests performed on remolded prestressed samples from the sand test section. In each test, the remolded sample was prestressed by cyclic loading prior to testing. Various combinations of intensity (15 to 30 psi), number of cycles (3 to 50) of prestress load, confining pressure during application of prestress load (variable as shown by curve 1, or constant 13 psi), and loading curves (curves 1 and 6) were used in attempts to duplicate the field stress-strain curve. Stress-strain curves developed from these tests approached duplication of the field stress-strain curve. A critical study of the data obtained in this series of tests indicated that further progress toward duplication of the field stress-strain curve might be accomplished by increasing the intensity of the prestress load and that approximately 10 cycles of prestress load appeared to be the optimum effort. It was therefore determined that the next series of tests would be conducted on samples prestressed with 10 cycles of 60-psi loading (maximum intensity of loading applied to field test section).

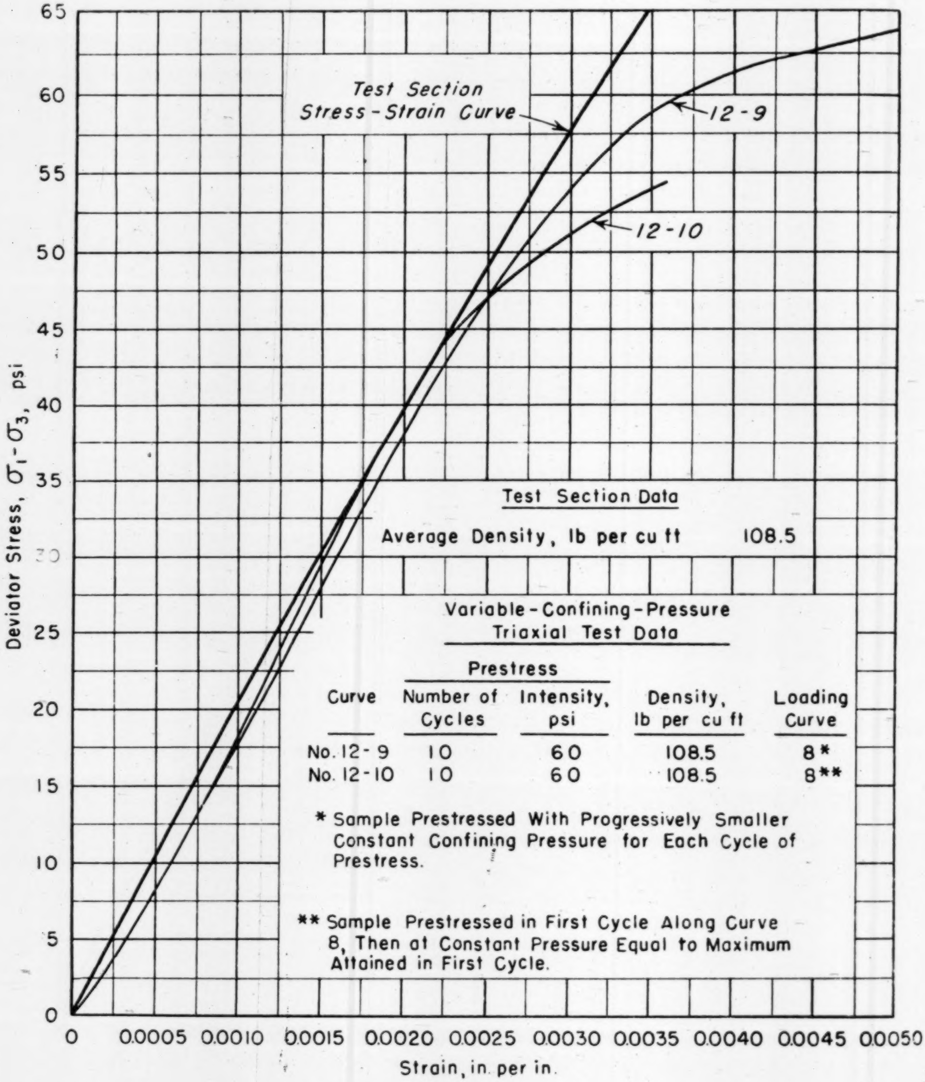


FIG. 5.—Comparison of Test Section Curve and Variable-Confining-Pressure Triaxial Test Curves from Remolded Sand Samples Prestressed at Various Loads.

The sixth series of tests consisted of 10 VCP triaxial tests performed on remolded samples from the sand test section. In each test, the sample was prestressed by 10 cycles of 60-psi prestress load prior to testing. The first six tests of this series were performed on samples confined by a constant pressure of 15 or 20 psi during application of the cyclic prestress load. Variable confining pressure during testing was determined through use of curve 6, Fig. 4. In four of these tests ($\sigma_3 = 15$ psi), the sample failed during application of the prestress loading. Stress-strain curves developed from two of the tests ($\sigma_3 = 20$ psi) approached duplication of the field stress-strain curve fairly well. The slope of these stress-strain curves was greater than that of the field stress-strain curve. Loading curve 7, Fig. 4, was selected for use in the seventh and eighth tests of this series. In the seventh test, it was intended to prestress and test the sample by loading along curve 7. This test was unsuccessful because the sample failed during application of the prestress loading.

A review of the testing procedure at this time indicated that duplication of the field stress-strain curve through use of curve 7 was dependent on selection of reasonable values for confining pressure to be used during application of the cyclic prestress loading. Experience had indicated that the value of confining pressure should be between 16 and 19 psi to insure completion of the test without failure of the sample. In the eighth test, the method of prestressing was changed. The cyclic prestress load was applied to the sample confined by a constant pressure (constant for each cycle) that was progressively and uniformly reduced for each cycle of prestress. The confining pressure varied from 20 psi for the first cycle to 16 psi for the tenth cycle of prestress loading. The prestressed sample was tested by loading along curve 7. The stress-strain curve developed from this test approached duplication of the field stress-strain curve.

Prior to completing the sixth series of tests, loading curve 8, Fig. 4, was drawn through a point indicating the average value (4 psi) for residual stress measured in the sand test section and parallel to curve 3. (This procedure prevents intersection of the loading curve with the Mohr failure envelope, produces a loading curve that will provide realistic loading condition at the beginning of the test, and eliminates failure regardless of magnitude of loading.) The last two tests in this series were made using loading curve 8. In the ninth test of this series, the cyclic prestress loading was applied in the same manner as in the eighth test, except that the confining pressure varied (in equal increments) between 18.25 psi for the first cycle and 16 psi for the tenth cycle of prestress load. This prestressed sample was tested by loading along curve 8. Stress-strain curve 12-9, Fig. 5, was developed from results of this triaxial test. The last test in this group was identical with the ninth, except for the manner of prestressing the sample. The first cycle of prestress load was applied by loading the sample along loading curve 8. The remaining cycles of prestress load were applied to the sample confined by a constant lateral pressure (18 psi) equal to the maximum lateral pressure attained during application of the first cycle of prestress. Stress-strain curve 12-10 was developed from the results of this test. Stress-strain curves 12-9 and 12-10 approach duplication of the field stress-strain curve so closely as to be identical for practical purposes.

DISCUSSION OF RESULTS

Test Method:

Through a process of trial and error, a method of testing small undisturbed samples cut from the homogeneous clayey-silt test section or remolded samples from the homogeneous sand test section was devised using the VCP triaxial tests which produced stress-strain curves that essentially duplicated the field stress-strain curves.

The VCP triaxial tests that provided the best results on the undisturbed clayey-silt samples are represented by stress-strain curves 5-*B* and 5-*G*, Fig. 3. During performance of these tests, the confining pressure was made to vary as shown by loading curves 7 and 9, Fig. 2. Curves representative of the field stress-strain curve were reproduced in six instances by use of these loading curves.

The VCP triaxial tests that produced the best results on remolded prestressed samples of sand are represented by stress-strain curves 12-9 and 12-10, Fig. 5. During performance of these tests (represented by curves 12-9 and 12-10), the confining pressure was made to vary as shown by loading curve 8, Fig. 4. Curves representative of the field stress-strain curve were obtained in each of these tests.

Loading Curves:

During performance of the VCP triaxial tests, the confining pressure was made to vary by loading the test sample along a loading curve. As previously discussed, the basic loading curves were developed from stresses measured 1 ft below the center of a single 1000-sq-in., uniform, circular load. The loading curves labeled 1 in Figs. 2 and 4 were developed from data obtained during tests performed on the homogeneous clayey-silt and sand test sections. In both cases, the curves represent the relation between the vertical less the horizontal pressure (deviator stress) resulting from applied surface loads and the horizontal pressure within the soil mass resulting from the applied surface loads. These loading curves show the variation of the lateral pressure within the soil mass caused by application of loads to the surface of the mass. However, since the curves represent total stresses, they do not pass through the origin; instead, they intersect the abscissa at a point determined by the residual and overburden stresses.

Attempts to measure residual stress during performance of tests on the homogeneous clayey-silt test section were not entirely successful. However, sufficient data were obtained to indicate that residual stresses existed within the soil mass. The test data presented in *Technical Memorandum No. 3-323* (Report 1)[3] indicate that the combined residual and overburden stress varied between 7 and 15 psi during testing of this section and averaged approximately 8 psi. The basic loading curve (curve 1, Fig. 2), which was used to establish guide loading curves for the VCP triaxial tests on the undisturbed samples of clayey silt, intersects the abscissa at a point determined by the overburden stress alone because reliable values for the residual stress were unknown. However, the loading curves that were used in the successful tests (curves 7 and 9, Fig. 2) intersected the abscissa at a point determined by the average value of the combined residual and overburden stress (8 psi).

Attempts to measure residual stresses in the homogeneous sand test section were more successful. The data obtained in tests on this section indicated that the combined residual and overburden stresses varied from 2 to 6 psi and averaged approximately 4 psi. The loading curve that was used in successful tests (curve 8, Fig. 4) intersects the abscissa at a point determined by this average value of the combined residual and overburden stresses.

Findings:

Perhaps the most important finding from this study is that the triaxial tests, including confined compression and cell tests as commonly performed in most laboratories, do not yield stress-strain relations representative of those effective in prototype soil structures.

The results of the triaxial tests indicate that there is a possibility of establishing a method of testing whereby the actual stress-strain relations existing within a large mass of homogeneous soil may be determined from small soil samples. VCP tests can be used to determine the true stress-strain relations at any point within a large homogeneous soil mass provided: (*a*) the soil structure within the test specimen approximately duplicates that within the soil mass, and (*b*) load is applied to the test specimen in the same manner that loads were applied at the point within the soil mass where the stress-strain relations are to be determined.

To assure that the soil structure within the test specimen is approximately the same as within the soil mass where the stress-strain relations are to be determined, undisturbed samples should be obtained at that point. In some instances, when dealing with cohesionless soils, it may not be possible to obtain undisturbed samples. In this case, remolded samples must be produced in the laboratory for testing purposes. It was shown in this test program that remolded samples of sand from the homogeneous sand test section could be prepared in the laboratory so that the soil structure within the test specimen was approximately the same as that in the test section. One fact determined during attempts to make remolded samples of sand with the same soil structure as in the test section is that density or strength cannot be used as a measure of duplication. The density of the remolded prestressed test specimen was determined before and after prestressing. The increase in density caused by prestressing was never greater than 0.2 per cent and averaged less than 0.1 per cent. Yet in all cases the modulus of deformation was considerably larger if the test specimen had been prestressed prior to testing. The angle of shearing resistance determined by standard triaxial tests performed on remolded prestressed samples of sand was the same as that determined from identical triaxial tests performed on remolded samples of sand that were not prestressed. The important fact here is that it would have been impossible to recognize which of the various remolded sand samples had the same soil structure as that of the homogeneous sand test section if the stress-strain relations existing within the test section had not been determined in advance.

When a test specimen having the desired soil structure has been obtained or prepared, the next problem is the selection of a realistic loading curve to use as a guide for applying the load to the test sample during performance of a triaxial test.

The results obtained from VCP triaxial tests performed on undisturbed clayey-silt samples indicate that triaxial loading must substantially duplicate that to be sustained in the prototype in order to develop realistic stress-strain curves from VCP triaxial tests performed on cohesive soil samples. The methods (described herein) used to develop curves 7 and 9 of Fig. 2 gave satisfactory results in the tests being reported.

The results obtained from VCP triaxial tests performed on remolded sand samples indicate that the curve representing failure (Mohr envelope) (curve 3, Fig. 4) can be used to develop realistic loading curves when the VCP triaxial test is to be performed on a cohesionless soil sample. In this triaxial testing program it has been shown that, provided the test specimen has approximately the same soil structure as that within a soil mass, approximate duplication of the field stress-strain curve can be accomplished by use of loading curve 8, Fig. 4. This loading curve is parallel to a curve representing failure (Mohr envelope) and intersects the abscissa at a point determined by an average value for the combined residual and overburden stresses. Here again the important fact is that it would have been impossible to determine which of the various loading curves represented the loading conditions within the homogeneous sand mass if the stress-strain relations existing within this mass had not been known in advance.

CONCLUSIONS

Based on the results of the triaxial testing program reported herein, the following conclusions appear valid:

1. The triaxial test as it is normally carried out does not yield stress-strain relations representative of those effective in prototype soil structures.

2. It appears that through proper preconditioning of specimens and special loading patterns, prototype stress-strain relations can be essentially duplicated in small laboratory tests.

3. The special loading conditions necessary are, primarily, use of a lateral loading varied in relation to the vertical loading similar to the way the horizontal and vertical stresses will be related in the soil mass represented, and, secondarily, inclusion of increments of overburden and residual stresses in the loading relation.

4. The special loading conditions do not appear to be critical for cohesionless materials, and theoretical relations and approximations likely will be adequate in establishing loading relations for these materials.

Conclusions more specifically related to the tests conducted and which appear worthy of note are as follows:

(*a*) The laboratory test methods developed will, when applied to tests on undisturbed samples from the homogeneous clayey-silt test section, yield stress-strain curves duplicating the field stress-strain curve for all practical purposes.

(*b*) The methods developed for laboratory preparation of samples of sand from the homogeneous sand test section to simulate conditions in the test section were satisfactory, since stress-strain curves for the laboratory-prepared samples duplicated the field stress-strain curve for all practical purposes.

(*c*) In prestressing a cohesionless soil:

(1) The magnitude of the prestress load is not critical but must be large enough to develop the proportional section of the stress-strain curve.

(2) The number of repetitions of the stress load is not critical provided at least 10 repetitions are used.

(3) The exact slope of the loading curve is not critical provided the curve does not approach the Mohr failure envelope.

(*d*) Prestressing the remolded sand samples caused a very small increase in density, no change in shearing strength, and a very large increase in the modulus of deformation.

DISCUSSION

MR. ROBERT F. BAKER.[1]—I have a question with reference to the figure showing a comparison between the theoretical and the measured deflections (the accompanying Fig. 6[2]). Did you get an edge shear at the side of the plate and was the amount of deflection resulting from such shear included in that recorded on the figure?

MR. RICHARD G. AHLVIN (*author*).—We did not determine deflections of the plate at the surface. The deflections in the figure are for a depth of 1 ft below the loading plate. They represent relative movement between a reference flange on the deflection gage and a reference rod in the ground beneath the test section.

MR. BAKER.—Did the deflection, as shown on the figure, rebound to zero on removal of the load?

MR. AHLVIN.—The deflections did not, in general, rebound to zero. There is an increment of permanent (nonrecovering) deformation resulting from each load application.

Figure 6 presents results from a study of which the work Mr. Brown and I are reporting is a part. Data from the parent study are being presented at the Fifth International Conference on Soil Mechanics and Foundation Engineering by W. J. Turnbull, A. A. Maxwell, and myself in a paper entitled, "Stresses and Deflections in Homogeneous Soil Masses." Also, a portion of these data were presented by C. R. Foster and S. M. Fergus in 1949 in Highway Research Board, *Research Report No. 12F*, entitled "Stress Distribution in a Homogeneous Soil."

MR. G. F. WEISSMANN.[3]—There is an interaction between the stress-strain relationship of the soil and the load-deflection characteristics of the measuring devices. Did the authors investigate the effect of this compliance of the pressure and deflection gages on the test results?

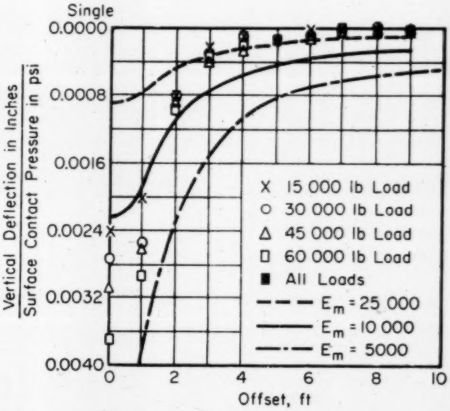


FIG. 6.—Deflections for a Depth of 1 ft Below the Loading Plate.

MR. AHLVIN.—Yes, we did. Some of the stress measurements in our homogeneous sand test section appeared to be impossibly large, and, as a result, we undertook a study of the pressure-cell - soil interaction. We have done a great deal of work on pressure-cell overregistration. Unfortunately the work has not been completed and reported and it is not now funded. Our studies indicate that over-registration in the sand will reach maximums normally less than about 10 per cent but sometimes as much as 25 or 30 per cent. These latter are always in zones of large stress obliquity where shearing deformations are occurring—zones at shallow depths and particularly near the edge of a loading plate.

---

[1] Professor of Civil Engineering, Ohio State University, Columbus, Ohio.

[2] The accompanying Fig. 6 was shown at the Session on Soils for Engineering Purposes but was not included in the authors' published paper.

[3] Member of the Technical Staff, Bell Telephone Laboratories, Inc., Murray Hill, N. J.

We had no way of making similar studies of our deflection gages. These were undesirably bulky units but they appeared to give good and reliable results in a number of installations.

MR. W. E. SCHMID.[4]—Were the stress-strain curves from the field that you were trying to match in the laboratory uniaxial stress-strain curves in the vertical direction? If not, how were they obtained?

MR. AHLVIN.—These are plots of the vertical minus the average lateral stress versus the vertical strain. The quantities compared are those whose ratio is equal to the modulus of elasticity in the equation relating vertical stress and strain for an ideal elastic system having a Poisson's ratio of 0.5.

MR. SCHMID.—I have one more question. How did you manipulate the lateral pressure? Did you apply air through a compressed air reducing valve?

MR. AHLVIN.—Tests were run using air as the confining fluid. Pressure was applied by adjustment of the air valve based on visual observation of pressure gage response. Loading curves of the type shown in our paper were followed by applying an increment of confining pressure followed by application of an increment of deviator load.

---

[4] Associate Professor of Civil Engineering, Princeton University, Princeton, N. J.

THE IMPORTANCE OF FREEZING RATE IN FROST ACTION IN SOILS*

BY E. PENNER[1]

SYNOPSIS

This study was concerned with determining the effect of rate of freezing on frost action under laboratory conditions. This was done for the saturated and near-saturated moisture conditions of the soil. The rate of freezing was measured both in terms of heat flow and rate of frost penetration. Three soils of widely differing properties were used to bring out major differences in behavior to freezing.

The results show a positive relation between net heat flow or frost penetration rate and heaving rate. Increasing the rate of heat flow away from the freezing plane in all cases increased the rate of moisture flow to the freezing plane and consequently also the heaving rate. This was shown to be true for the three soils studied at high moisture contents.

It is not correct to compare the frost susceptibility of different soils in laboratory freezing experiments on the basis of equal rates of frost penetration when, in fact, this does not occur in the field. The rate of heat extraction used in laboratory tests of frost heaving should be related to the actual heat flow in the environment of the soil.

The results presented should be useful in setting up practical laboratory freezing procedures for the realistic evaluation of the frost susceptibility of soils in the field.

With the commonly used frost action criteria based on grain-size distribution, it is not always possible under field conditions to predict the frost susceptibility of all soil materials (**1**).[2] As an alternative, the field performance of these soils might be determined from a laboratory freezing experiment, which approach is now fairly common practice in soil engineering.

Resorting to such performance freezing experiments is an admission that knowledge of the physicochemical characteristics of soil is not yet sufficiently advanced to allow prediction of frost susceptibility. This is particularly true of soils which are borderline with respect to grain-size criteria when both freezing temperatures and high-moisture conditions exist.

The most pressing problem is the evaluation of the degree of frost susceptibility by a relatively simple and meaningful laboratory freezing experiment. In the long run, however, the aim in soil engineering is to develop a better understanding of the soil characteristics that make a potentially frost heaving soil.

Two of the most important conditions that influence the results of a laboratory freezing experiment are the rate at which the specimen is frozen and the water supply. The differences between these conditions imposed on a soil specimen in the laboratory as against those that occur naturally in the field are thought to account for the unsatisfactory results often obtained in the past.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 27–July 1, 1960.

[1] Research Officer, Soil Mechanics Section, Division of Building Research, National Research Council, Ottawa, Canada.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper.

The research reported here is an attempt to formulate a more realistic basis for laboratory freezing experiments. The main variable studied was the rate of freezing at constant-moisture conditions external to the specimen. To a lesser extent, the second variable (water supply) was studied, but much further work is necessary in the unsaturated state, particularly because of the interrelationship between unsaturated permeability and moisture content. Three soils with widely different properties were used to show major differences, if any, in behavior to freezing.

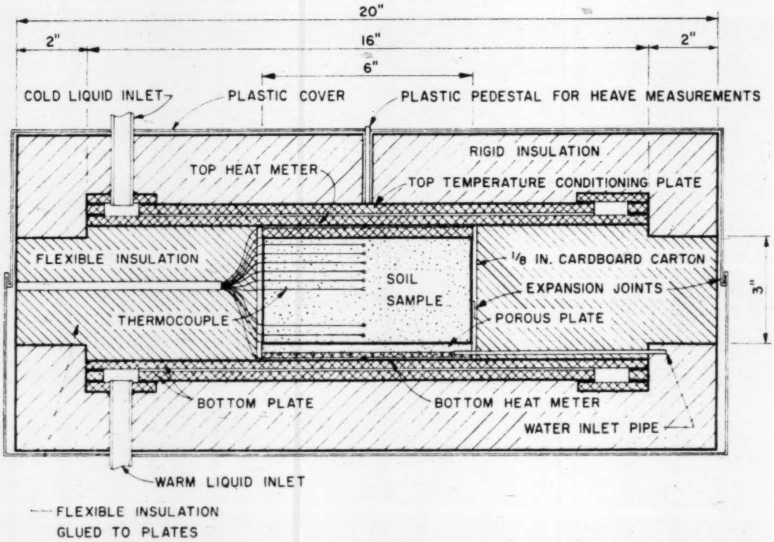

20"
2"
16"
2"
6"
COLD LIQUID INLET
PLASTIC COVER
PLASTIC PEDESTAL FOR HEAVE MEASUREMENTS
RIGID INSULATION
TOP HEAT METER
TOP TEMPERATURE CONDITIONING PLATE
FLEXIBLE INSULATION
SOIL SAMPLE
1/8 IN. CARDBOARD CARTON
EXPANSION JOINTS
POROUS PLATE
THERMOCOUPLE
3"
BOTTOM PLATE
BOTTOM HEAT METER
WATER INLET PIPE
WARM LIQUID INLET
FLEXIBLE INSULATION GLUED TO PLATES

FIG. 1.—Frost Cell MK II.

REVIEW OF PREVIOUS WORK

Beskow's early work suggested that the rate of heaving was independent of the rate of freezing (2). After further experiments, he stated "the tests have fully proved, as the previous tests did not fully show, that for a constant pressure the rate of heaving is independent of the rate of freezing." He further stated, "it should be noted, however, that this is completely valid only for relatively permeable soil." Beskow did recognize that for all soils under conditions where the frost line was stationary various rates of heave were possible and dependent on the heat flow, but this did not hold for a penetrating frost line.

The U. S. Army Corps of Engineers state, "rate of heave has been found to be relatively independent of rate of freezing over a range of freezing rates employed in the investigation" (3). This is the basis for the frost susceptibility classification that holds for freezing rates between $\frac{1}{4}$ to $\frac{3}{4}$ in. frost penetration per day.

In recent investigations on the effects of freezing rate on heaving rate for one soil, Higashi (4) indicates that heaving rate varies inversely as the freezing rate according to the expression:

$$d\xi/dt = A - B(dx/dt)$$

where:

$d\xi/dt$ is the rate of heaving,

dx/dt is the rate of frost penetration, and

A and B are constants depending on the original density.

The findings by Higashi are in complete contradiction to results obtained by all other research workers to date.

Previous experiments carried out by the author indicated there was a strong positive correlation between the rate of heat removal and rate of heaving (5).

METHODS AND MATERIALS

Description of Frost Cell:

The frost cell (Figs. 1 and 2) consisted of two large temperature-conditioned metal plates that determined the temperatures at the top and bottom of a 3 in. long soil specimen. The 6-in. diameter specimen was contained in a wax-impregnated cardboard cylinder closed at the upper end by a metal plate and at the lower end by a porous plate. The porous plate was uniformly supplied with water through a spiral groove system in a metal backing plate.

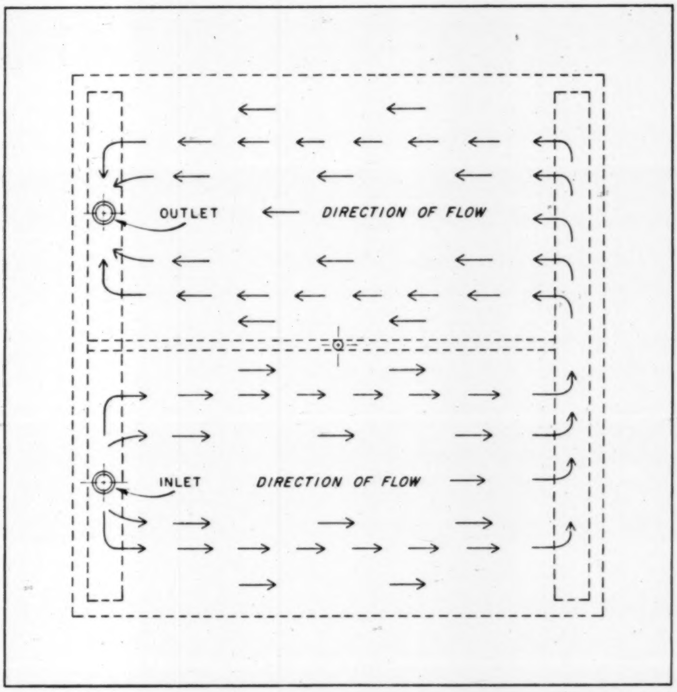


FIG. 2.—Plan Showing Flow Path in Cooling Plates of Frost Cell.

A Gier and Dunkle heat transducer, embedded in an epoxy resin slab $\frac{1}{4}$ in. thick, was placed at each end of the specimen holder. The heat meters were calibrated and checked periodically by imposing a known temperature gradient across a 2-in. hard rubber slab for which the thermal conductivity coefficient was accurately known. In the calibrating system, the heat meters were also placed at each end of the rubber slab. Calibration was carried out in the same temperature range as the freezing experiment.

The temperatures, at known positions in the soil specimen, were measured using 30-gage constantan-copper thermocouples. The thermocouple points were positioned at various elevations along the center line of the 3 in. long specimen. The holes were drilled at right angles to the heat flow direction using a long, thin bit in a drill press. Thermocouple and heat meter measurements were recorded with a specially modified high-precision millivolt recorder.

The cylindrical specimen was surrounded by 5 in. of glass fiber insulation. This appeared to be sufficient to prevent any appreciable thermal disturbance from the outside. To facilitate heaving and still provide insulation from top to bottom, the glass fiber insulation was glued to the top and bottom plates with ordinary rubber cement. As the soil samples heaved, the "accordion" action of the insulation provided continuous insulation from outside thermal disturbances.

The cardboard sample holder was provided with a lap joint one third the distance from the bottom to allow heaving at a very low friction between container and specimen. The joint was sealed with grease to prevent moisture loss as were the holes in the sample holder for the thermocouples. The system as a whole was checked thermally using a number of different heat and moisture flow balances which are described later.

The plates were temperature conditioned by circulating ethylene-glycol mixtures controlled to within ±0.05 C. The closeness of the temperature control was considered adequate, as no variation in heat meter output was created by the off-on cycles of heating and cooling. This temperature controlling apparatus has been described in more detail in a previous paper (6).

Constant Water Head Device:

Water was supplied to the specimen from the outside by a plastic tube that fed the demands of the porous plate through a spiral groove system. The water used by the specimen was supplied at a constant head with an automatic balance especially constructed and accurate within ±0.01 ml.

The balance consisted of an analytical balance which had one end of the beam fixed through a spring to the balance floor. Hung on the other end of the beam was a plastic water container with a bubbler hole. A soft rubber hose was attached to the container for the water outlet. A variable differential transformer core that was fixed above the spring was adjusted to move not more than ±0.1 in. from the horizontal beam position. The electromotive force output from the secondary was demodulated and recorded on a standard millivolt recorder. The balance was calibrated periodically and proved to be reliable. The water loss per unit deflection was linear over the complete range of beam displacement.

Description of Soil:

The three soil materials selected were of widely differing properties. The clay sample was from the Ottawa Valley marine clay. Both its geology and geotechnical properties have been described by Eden and Crawford (7). A block sample, approximately 1 cu ft in volume, was procured from a depth of 16 ft, which was well below the brown oxidized zone but within the fissured crust.

The soil designated as PFRA silt was procured by The Prairie Farm Rehabilitation Administration from the basement of a partially constructed house in Saskatoon, Sask., which had suffered severe frost action when left unheated during one winter.

The coarsest material was supplied by the Ontario Department of Highways and was obtained from a gravel pit near

Lindsay, Ont. This material was put through a No. 8 sieve to remove any stones.

The grain-size distribution curves for these soils, shown in Fig. 3, were determined by means of hydrometer analysis. For reference purposes in this report, these soils will be designated as Leda clay for the heaviest soil, PFRA silt for the intermediate soil, and Lindsay sand for the coarsest soil. According to the Massachusetts Institute of Technology classification, the percentage clay, silt, and sand are as given in Table I.

TABLE I.—CLAY, SILT, AND SAND FRACTIONS FOR THE SOILS USED.

	Clay, per cent	Silt, per cent	Sand, per cent
Leda clay	64	36	...
PFRA silt	9	43	48
Lindsay sand	7	13	80

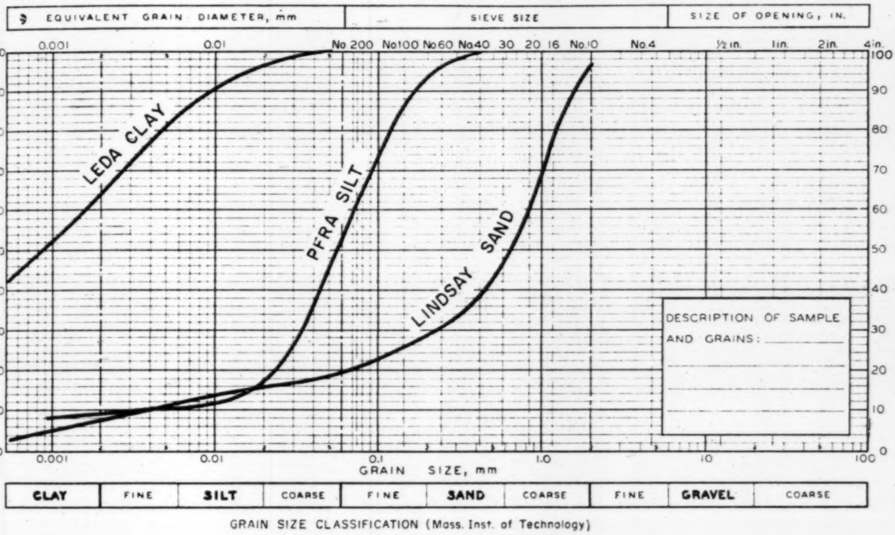


FIG. 3.—Mechanical Analysis of Soils.

Soil Specimen Preparation:

The specimens of Lindsay sand and PFRA silt were prepared with an automatic compaction apparatus in a 6-in. diameter mold which had been cut down in height from 5 in. to 3 in. These soils were first air-dried and thoroughly mixed to obtain uniformity. The moisture content at maximum dry density was determined using the modified AASHO procedure (8). All the specimens of Lindsay sand and PFRA silt were prepared at this determined maximum density. Following compaction, the samples were placed in a pressure-vented oven at 105 C and left for 48 hr. Very little shrinkage in either soil occurred on drying.

The water saturating procedure was as follows. The oven-dry sample was placed in a vacuum desiccator and continually evacuated for 24 hr. Following this, vacuum deaired water was introduced into the desiccator under vacuum conditions. The sample was allowed to remain in contact with water for at least 24 hr. The dry densities for the Lindsay sand samples were between 2.19 and 2.21 g per cu cm (136.5 to 138.5 lb per cu ft) and for the PFRA silt 1.76 to 1.77 g per cu cm (110 to 110.5 lb per cu ft).

The moisture content achieved with this saturating technique ranged from 7.96 to 8.46 per cent for the Lindsay sand and 18.9 to 19.5 per cent for the PFRA silt.

The Leda clay specimens were prepared differently because compaction difficulties were anticipated and large attendant shrinkage would occur on drying. Once air- or oven-dried in a block sample, resaturating the clay with any sort of reproducibility would be time-consuming and difficult. The clay was procured in an undisturbed block, and since considerable grain-size and moisture-content variations were shown to occur throughout, the sample was thoroughly puddled at its natural moisture content using a paddle-type paint mixer. Liquefaction was easily achieved since the liquidity index was 1.2 (**7**).

The semifluid mix was then poured into the 6-in. diameter compaction mold to a height of 3 in. and consolidated under static pressures in progressive steps to a maximum of 6 kg per sq cm. The densities achieved in this way ranged from 1.44 to 1.47 g per cu cm (90 to 91.7 lb per cu ft) with a moisture content range from 32.4 to 33.9 per cent. From this consolidated material, a sample 2 in. high was cut and directly placed into the frost cell.

Ice Content Determinations for Frozen Soil:

For one soil (Lindsay sand), the relation between temperature and ice content was determined. These determinations were necessary to check the heat flow quantities indicated by the heat meters in the case of frost heaving in a closed system. How these values were used will become evident below.

The samples were compacted to the same density as in the frost-heaving experiment. This was done by increasing the compactive effort in the Harvard compaction apparatus until the desired density was achieved. The moisture content used in the preparation of these small specimens was the same as that previously determined at the maximum density. The saturating procedure was also the same as described previously.

Samples were frozen at various temperatures at the bottom of a 2-gal well inside a tank under closely controlled temperatures. (This tank has already been described (**9**).) The temperature variations between samples in the well at any one time were usually as close as could be read on the recorder chart except for one case where a difference of 0.05 C occurred.

The heat released was determined calorimetrically in a 1-qt thermo flask using a glass stirrer. The water equivalent, *E*, of the calorimeter was determined by melting ice cubes in the same temperature range in which the ice content determinations of the soil specimens were made. An average value for *E*, 29.85 cal per deg Cent, was based on six separate determinations: 29.29, 28.12, 30.26, 30.70, 30.73, and 29.98. The statistical standard error is ±0.41 cal per deg Cent.

Temperature changes occurring in the calorimeter were evaluated according to the method described by Lovell (**10**). This allowed for the different heat loss rates of the calorimeter fluid before frozen soil or ice was introduced and after it had thawed. The temperature was continuously measured with a curve-drawing millivolt recorder. The equal area measurements were done on the recorder chart using a simple polar-planimeter.

Values for the specific heats of water, ice, and the latent heat of fusion of ice were taken from the literature. The value of 0.17 for the specific heat of the soil was taken from results determined by

TABLE II.—TEMPERATURE GRADIENTS AND THERMAL CONDUCTIVITIES PRIOR TO INDUCING ICE CRYSTALLIZATION.

Soil	Gradient		Thermal Conductivity	
	deg Fahr per in.	deg Cent per cm.	Btu in. per hr sq ft deg Fahr	cal per hr cm deg Cent
Lindsay sand (density 2.19 to 2.21 g per cu cm)......	1.03	0.22	12	14.9
	0.94	0.21	13.9	17.2
	0.95	0.21	12.6	15.6
	1.04	0.23	12.9	16.0
	1.04	0.23	13.1	16.2
	0.90	0.20	12.7	15.8
Average.........	0.98	...	12.9	16.0
PFRA silt (density 1.76 to 1.77 g per cu cm)...........	0.96	0.21	13.0	16.1
	1.19	0.26	12.4	15.4
	1.19	0.26	14.3	17.7
Average.........	1.11	...	13.2	16.4
Leda clay (density 1.44 to 1.47 g per cu cm)...........	1.85	0.40	6.5	8.0
	1.75	0.38	6.4	7.9
	1.89	0.41	6.3	7.8
	1.60	0.35	6.9	8.5
Average.........	1.77	...	6.5	8.1

TABLE III.— A COMPARISON BETWEEN NET HEAT FLOW AS MEASURED BY THE HEAT METERS WITH THE LATENT HEAT OF CRYSTALLIZATION OF THE WATER FROZEN DURING THE SAME PERIOD.

Period	Heat Meters, Btu $(Q_T - q_a)$	Moisture Flow, Btu $(q_e + q_f)$
1 to 2............	15.20	12.40
2 to 3............	13.55	12.26
3 to 4	10.50	9.86
4 to 5............	8.85	8.86
5 to 6............	7.05	6.83
6 to 7............	6.74	6.65

Kersten (11) for a similar soil in the same temperature range.

Freezing Procedures for Heaving Experiments:

After the specimen was placed in the frost cell, the porous plate was connected to the outside water supply (except in the case of closed systems) and a predetermined temperature gradient was imposed across the specimen with the temperature at the top near 0 C. When the heat flow into and out of the sample were the same within the limits of error, freezing was started by first lowering the temperature of the cold end of the soil slightly below freezing and inducing crystallization with a thin wire. The prefreezing thermal conductivities and temperature gradients for the soils studied are given in Table II. These values are for the saturated condition and density previously described.

RESULTS AND DISCUSSIONS

Discussions of Heat Flow and Moisture Balance:

The amount of heat, Q_T, flowing out of the freezing soil system (over a given period of time) while frost heaving and penetration of the frost line is occurring is the sum of the following quantities:

q_a = heat entering specimen,

q_b = heat removed in cooling of wet unfrozen soil,

q_c = heat removed in cooling of ice and soil above ice lens,

q_d = latent heat released on freezing of *in-situ* water,

q_e = latent heat released on freezing water moved to freezing plane, and

q_f = cooling of moisture during movement along the temperature gradient in unfrozen soil.

That is,

$$Q_T = q_a + q_b + q_c + q_d + q_e + q_f \;..\;(1)$$

A reduction in the rate of frost-line penetration reduces the values of q_b, q_c, and q_d. When the rate of frost-line penetration is zero and the moisture flow is small, q_b, q_c, and q_d approach zero, and:

$$Q_T = q_a + q_e + q_f$$

or, by transposing,

$$Q_T - q_a = q_e + q_f \;........\;(2)$$

Both Eqs 1 and 2 describe heat flows in the unsteady state. Equation 2 describes a condition where the length of the frozen portion is increasing due to the freezing out of water that has moved to the freezing plane. When the flow of water is small, a quasi-equilibrium or nearly steady-state condition is achieved.

In an open system, balancing the difference between heat-in and heat-out against moisture flow provides an approximate check on the operation of the apparatus. This was done a number of times. The results of one such trial are given in Table III for the condition of temperature distribution given in Fig. 4 for the periods marked from 1 to 7 inclusive. Each period is approximately 24 hr long. The periods between 4 and 7 approximate the conditions of Eq 2.

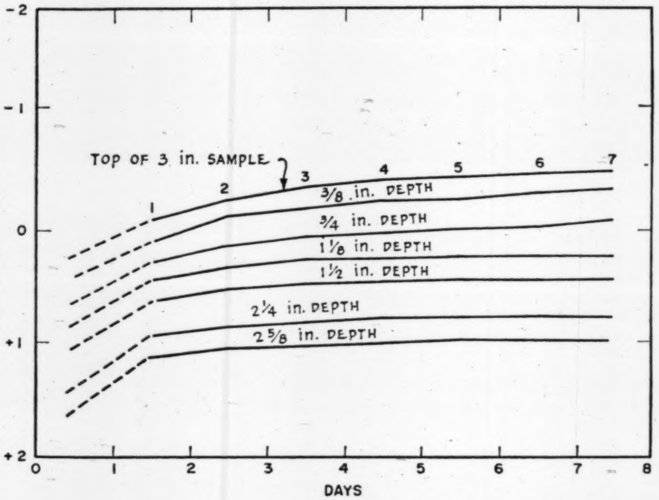


FIG. 4.—Temperature Distribution versus Time at Various Positions in the Specimen.
NOTE.—Numbers indicate periods used for heat-flow and moisture-flow comparisons.

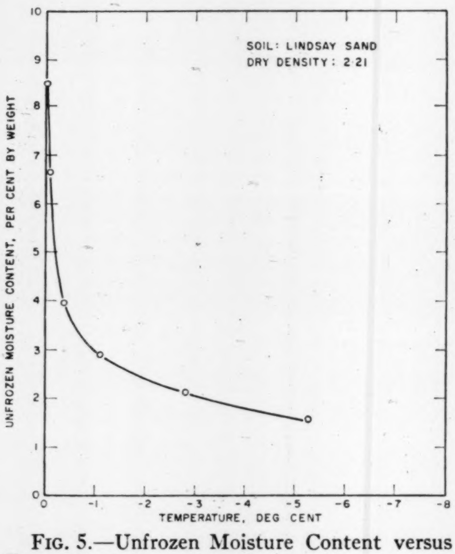


FIG. 5.—Unfrozen Moisture Content versus Temperature.

When no moisture is allowed to flow into the sample after freezing starts and the initial and final temperature gradients are known together with the amount of unfrozen water as a function of the temperature, another check on the functioning of the system is possible. In this case $Q_T - q_a = q_b + q_c + q_d + q_e + q_f$. Such a check was made on a specimen of Lindsay sand between the limits of initial and final temperature gradients.

From the curve of unfrozen water

content as a function of temperature given in Fig. 5, together with a knowledge of the specific heat of sand, the balance given in Fig. 6 was obtained. The curve is a cumulative plot of $Q_T - q_a$ based on results obtained from the heat meters. To the right side of the graph the value $A = 52.32$ Btu is the total value of $q_b + q_c + q_d + q_e + q_f$. This value, although 4 per cent higher than that based on the heat meters, is considered a fairly good balance in view of the many difficulties involved in such an experiment.

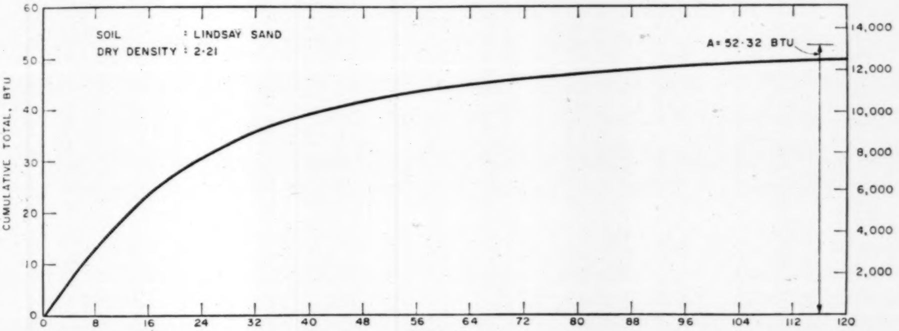


FIG. 6.—Net Difference Between Heat Out and Heat In as a Function of Time in a Closed System.

Numerous checks on the functioning of the frost cell similar to those already mentioned were carried out from time to time. Rechecking the calibration of the heat meters was also carried out periodically as a means of maintaining the best possible values for the heat-meter constants.

Heaving Rate Experiments:

Series 1.—To determine the effect of varying the rate of heat extraction on the rate of moisture flow to the frost line, two series of experiments were carried out. In the first series, after crystallization had been induced, the temperature of the top plate was set at a given temperature below freezing and the frost line was allowed to propagate down without any further change in the cold-side temperature. The water table was kept level with the bottom of the specimen. Temperatures, heat flow, and moisture movement in to the sample were continuously recorded. In the next run, with a new specimen, the cold-side temperature was set slightly lower, thus inducing a faster frost-line penetration and also a more rapid heat extraction rate. In this way a number of different rates of freezing were carried out for each soil.

The results of the heat-flow and moisture-flow measurements are given in Fig. 7 for the three soils. The curves with the circles are the cumulative difference between heat flow out of the sample and heat flow into the sample in terms of Btu's or calories. The curves with the dots give the moisture flow in to the sample as demanded by the freezing front. This is expressed in terms of its latent heat in Btu's when frozen, 80 cal per g or 144 Btu per lb.

The obvious conclusion from these results is that an increase in the rate of heat extraction from the sample increases the moisture flow into the specimen and, consequently, its heaving rate is increased. This was true for all three soils, but it must be remembered that the external water source was at the level of the base of the specimen; that is, saturation or near-saturation conditions prevailed.

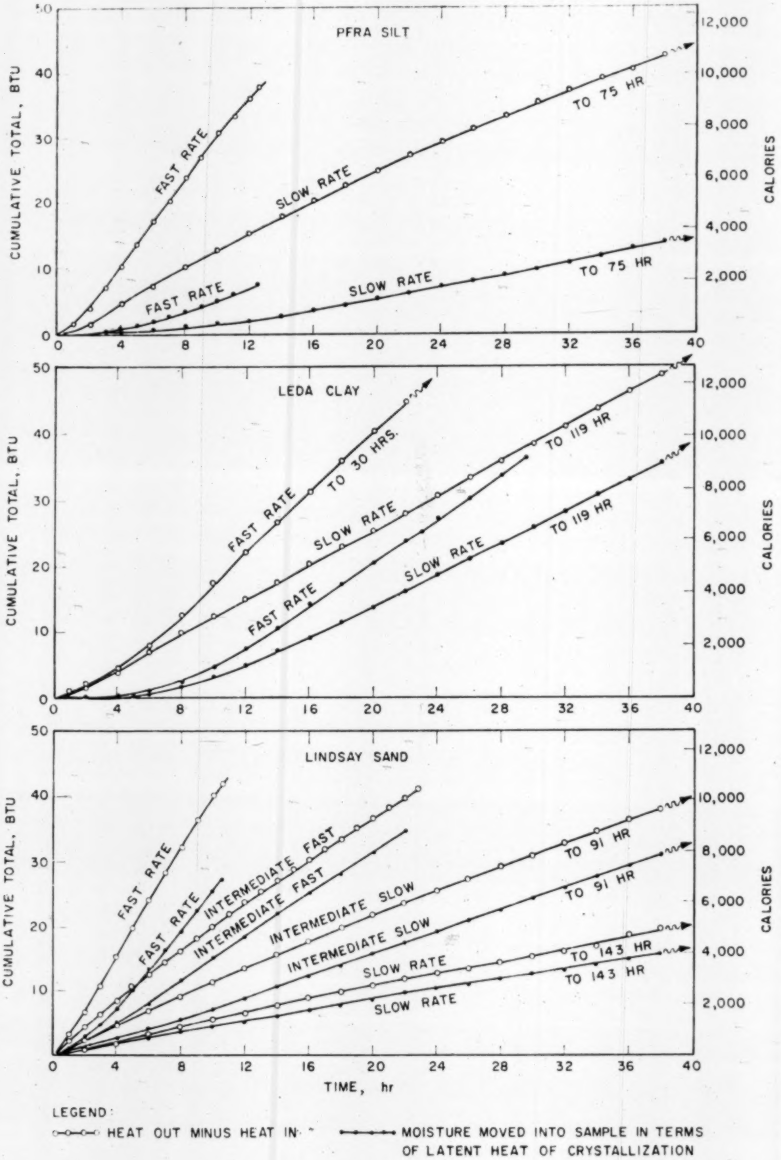


FIG. 7.—Cumulative Value of Net Heat Flow and Moisture Flow versus Time.

The results of these experiments are summarized in Fig. 8. The points on the graph are the average rate of moisture flow into the sample for an average rate of heat extracted. Again, the heat-flow parameter is heat flow out of the sample minus heat flow into the sample. On the

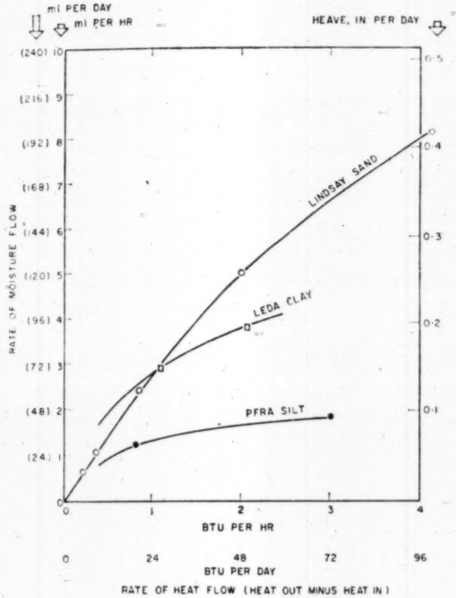


FIG. 8.—Moisture Flow and Heave versus Net Heat Flow.

NOTE.—1. By separate experiment for each rate and for each soil. 2. Water table at base of specimen.

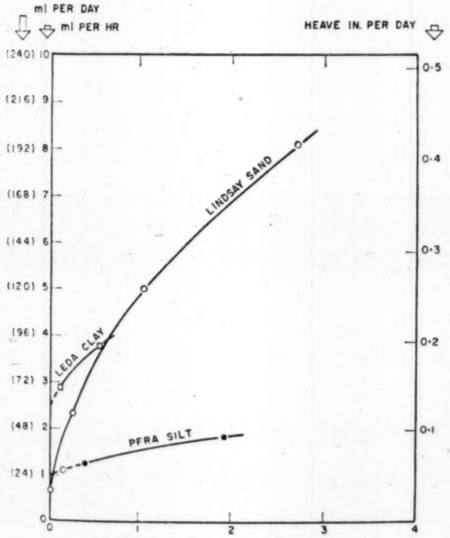


FIG. 9.—Rates Moisture Flow and Heave versus Frost Penetration.

right side of Fig. 8 are given the rates of heave in terms of inches per day. These curves must, of course, go to zero, that is, when heat flow out of minus heat flow into the sample is zero, no ice segregation can occur. This is theoretically the case and is shown experimentally for Lindsay sand in Fig. 8. In the other soils, the plotted points have been connected by curves pointing to zero. There appears to be no question that the heave rate is closely related to the net heat flow, although there is some dropping off in the amount of water that flows to the freezing plane per unit heat extracted as the rate of net heat flow is increased.

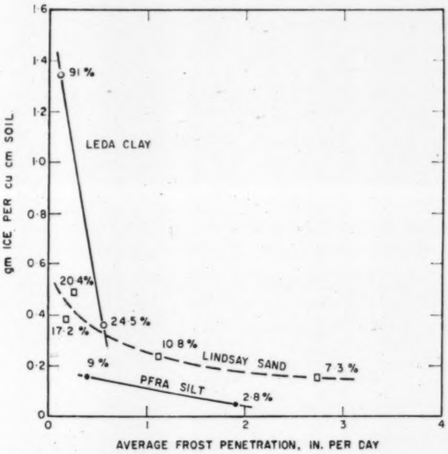


FIG. 10.—Average Amount of Ice Accumulation per Unit Original Volume of Soil versus Frost Line Penetration.

NOTE.—Numbers indicate per cent moisture content by weight added by the frost action process (g water per g dry soil).

Figure 9 is a plot of the average rate of frost-line penetration per day versus rate of heave and moisture flow. Frost-line penetration was determined by dividing the measured frozen layer at the end of the experiment by the time. If the curves in Fig. 9 are extrapolated to zero frost penetration, Eq 2 is approximately applicable and represents frost heaving without frost-line penetration. For Lindsay sand, this was determined experimentally as is shown by the plotted point.

When the frost-line penetration has stopped, heave rate does not stay constant in a freezing system such as has been described here. As the ice lens grows, the thickness over which the temperature difference is applied increases and so reduces the temperature gradient. This situation is true unless the temperature on the cold side is reduced. If this is not done, the heaving rate gradually decreases. This is shown in Table III and is also evident in many of the graphs.

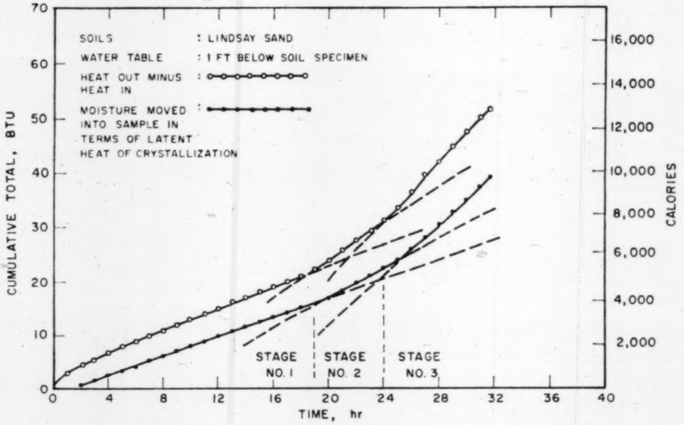


FIG. 11.—Cumulative Values of Net Heat Flow and Moisture Flow versus Time.

The average amount of ice accumulated per unit volume of original soil also varies with the rate of frost-line penetration. The results shown in Fig. 10 indicate that the amount of ice per unit volume of soil varies inversely to the rate of frost-line penetration. The numbers beside the plotted points are the average moisture contents of the frozen layer. These figures do not include the original water content of the soil but merely that moved in due to the frost action process.

This has obvious implications when applied to spring break-up under field conditions and will be discussed later. The parameter, grams of ice per cu cm of soil frozen, becomes infinitely large as the rate of frost-line penetration approaches zero and has no real meaning when the frost line becomes stationary. The greatest accumulation of ice occurred in Leda clays at the slowest rate when more than 50 per cent of the volume in the frozen state consisted of ice frozen out from moisture moved in the specimen.

Series 2.—In the second series of experiments on heaving rates, the water table was dropped 12 in. below the bottom of the specimen rather than level with the bottom of the specimen as in the first series. Following this, the temperature gradient was applied with the cold end of the soil close to zero. When heat into and heat out of the sample were equal, the cold end of the soil was supercooled. Crystallization was induced and the temperature of the cold plate was set to a predetermined value. After allowing the system to run for 20 to 22 hr, the cold-side temperature was further reduced. Sometimes the temperature on the cold side was lowered a third time, resulting in three distinct stages of cooling rates.

The cumulative difference between

heat flow out of the sample and heat flow into the sample as measured with the heat meters is shown for one soil in Fig. 11. The moisture flow into the sample is again shown in terms of Btu's based on the latent heat of fusion.

In all cases when the rate of heat extracted from the sample was increased, an increase in moisture flow resulted. The summary results are shown in Fig. 12. The similarity between results given in Fig. 8 and Fig. 12 are striking. With these soils at the densities given, lowering the water table by 12 in. had very little effect on the heaving rate. This means that the unsaturated permeability was not limiting frost heaving.

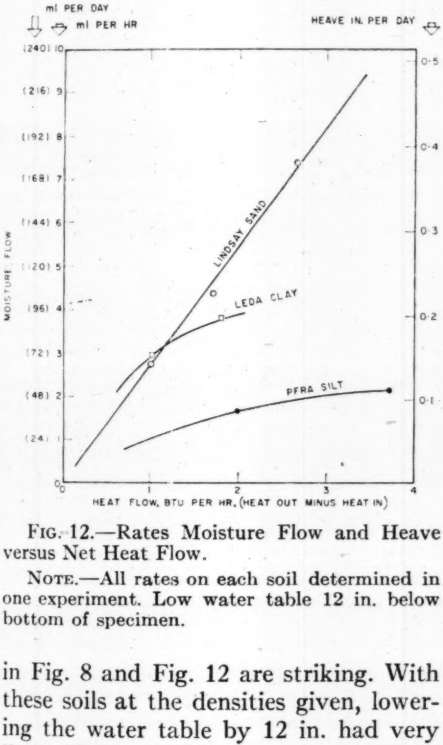


FIG. 12.—Rates Moisture Flow and Heave versus Net Heat Flow.

NOTE.—All rates on each soil determined in one experiment. Low water table 12 in. below bottom of specimen.

GENERAL DISCUSSION

Dependence of Ice Distribution on Rate of Freezing:

Results of these experiments show how different rates might influence the severity of spring break-up. A slow penetration of the frost line gives a higher concentration of ice in the frozen layer than a fast penetration. More water would be released per unit volume of soil in the spring in a year when the frost-line penetration was slow. The implication is that softer conditions might result in the road during the thawing period if other factors, such as drainage and rate of thaw, were equal. This is, however, only one aspect of frost action damage.

When frost heaving is the destructive element as opposed to loss of strength, the situation is reversed: the faster the rate of freezing, the greater the total amount of heave for a given period.

Frost Heaving in the Field, Simulated in the Laboratory:

Soil freezing and the associated ice segregation process are closely related to the heat flow phenomenon. The experimental evidence presented here indicates that it is misleading to compare the frost susceptibility of different soils in a laboratory freezing experiment where the frost-line penetration rate is the same. Indeed, a knowledge of temperature changes in different soils exposed to the same climate leads to the same conclusion. What in fact determines the depth of frost penetration is the rate of heat removal, the water supply, the frost susceptibility of the soil, the *in-situ* water content, etc.

The field work done recently by the Division of Building Research with regard to heat flow into and out of the ground at various seasons provides pertinent information that may well serve as a guide for establishing the rate of cooling in laboratory freezing experiments for the Ottawa area. Obviously, this will vary depending on the climate in a particular area.

Gold (12) found the average heat flow from the ground in the winter at one site in the Ottawa area to vary considerably during two winters of observation. In the period Dec. 10, 1955, to Jan. 26, 1956, and Jan. 26 to April 3, 1956, the average heat flow was 27 and 5.7 cal per sq cm per day respectively. In the period Nov. 5, 1956, to Jan. 4, 1957, and Jan. 4 to March 7, 1957, the corresponding values were 27.6 and 17.9 cal per sq cm per day. He concluded the average heat flow from the ground in the Ottawa area to be of the order of 20 cal per sq cm per day. Further results published by Pearce and Gold (13) show the measured heat-flow rate from January to early March, 1958, to be about the same on the average. Information such as this can be found in the literature for various parts of the world.

Soil temperature changes due to diurnal variations or cold spells in the weather decrease with depth. Therefore, the heat flow at the frost plane will fluctuate considerably more near the surface. As the frost line penetrates, these fluctuations will be attenuated to some extent depending on the depth. Simulating such fluctuations in the laboratory may not prove to be necessary; at the same time, applying an average rate of heat removal has its shortcomings.

The greatest heaving rates are usually reported during freezing-and-thawing periods. This has led some workers to believe a fluctuating frost line to be a necessary prerequisite to ice lens formations. Since these are periods of greatest heat flow with the frost line near the surface, they are probably also periods of greatest ice lensing activity if the moisture contents are high. It is noted here that if the rate of ice segregation was not related to the rate of heat extraction, the rapid growth of spring frost boils would not be possible. It is incorrect to compare the frost susceptibility of two different soils on the basis of equal rates of frost penetration; using the same rate of heat extraction is the more realistic basis.

SUMMARY AND CONCLUSIONS

The results presented in this paper, which apply to saturated or near-saturated conditions, show the strong influence of net heat flow from the soil on the heaving rate. Increasing the rate of heat flow away from the freezing plane in all cases increased the rate of moisture flow and, consequently, the heaving rate. This was shown to hold for three soils of widely different properties.

The average concentration of ice in the frozen soil, resulting from water moved to the freezing zone, increased as the rate of freezing was decreased for all soils.

For these three soils at the densities used, the proximity of the water table was not a critical factor. Lowering the water table by 12 in. made no essential difference to the frost heaving rate; therefore, the unsaturated permeability did not limit frost heaving.

At the slower rates of freezing, a larger proportion of the net heat extracted from the specimen was derived from the latent heat of crystallization.

It is unsatisfactory to compare the frost susceptibility of different soils in laboratory freezing experiments on the basis of equal rates of frost penetration because the same amount of heat extracted for two different soils does not produce the same frost penetration.

The rate of heat extraction used in the laboratory should be related to the actual heat flow in the environment in which the soil exists. Finally, it is believed these results are useful in setting up practical laboratory freezing procedures for the realistic evaluation of the frost susceptibility of soils in the field.

Acknowledgments:

The author wishes to acknowledge the assistance of his colleagues in the Division of Building Research and in particular that of D. Eldred of the technical staff in operating the frost cell equipment. The author is also grateful for the encouragement and continued interest of the Director, R. F. Legget, and Assistant Director, N. B. Hutcheon in frost action research. This is a contribution from the Division of Building Research, National Research Council (Canada) and is published with the approval of the Director of the Division.

REFERENCES

(1) R. M. Hardy, Discussion in Symposium on Frost Action, *Proceedings*, 38th Convention, Canadian Good Roads Assn., Sept. 25–27, 1957, pp. 260–261.

(2) G. Beskow, "Soil Freezing and Frost Heaving with Special Application to Roads and Railroads (1935)," The Swedish Geological Society, Series C, No. 375, 26th year, Book No. 3. Translated by J. O. Osterburg. Published by the Technological Institute, Northwestern University, Evanston, Illinois, pp. 14–21 (1947).

(3) "Cold Room Studies—Third Interim Report of Investigations," *Technical Report No. 43*, U. S. Army, Corps of Engineers, Oct. 1958.

(4) A. Higashi, "Experimental Study of Frost Heaving," *Research Report No. 45*, U. S. Army, Corp of Engineers, Snow, Ice and Permafrost Research Establishment, Aug. 1958.

(5) E. Penner, "The Mechanism of Frost Heaving in Soils," *Bulletin No. 225*, Highway Research Board, Jan. 1959, pp. 1–13.

(6) E. Penner, "Pressures Developed in a Porous Granular System as a Result of Ice Segregation," *Special Report No. 40*, Highway Research Board, pp. 191–199 (1958).

(7) W. J. Eden and C. B. Crawford, "Geotechnical Properties of Leda Clay in the Ottawa Area," *Proceedings*, Fourth International Conference on Soil Mechanics and Foundation Engineering, Vol. 1, Division 1, pp. 22–27 (1957).

(8) T. W. Lambe, "Soil Testing for Engineers," Mass. Institute of Technology, John Wiley & Sons Inc., New York, N. Y. (1951).

(9) E. Penner and P. Robillard, "The Use of Waste Sulfite Liquor to Reduce Frost Heaving in Soils," *Transactions*, Engineering Inst. Canada, Vol. 3, No. 4, Dec. 1959, pp. 107–109.

(10) C. W. Lovell, "Temperature Effects on Phase Composition and Strength of Partially Frozen Soil," *Bulletin No. 168*, Highway Research Board, pp. 74–96 (1957).

(11) M. S. Kersten, "Laboratory Research for the Determination of Thermal Properties of Soils," *Research Laboratory Investigations*, Engineering Experiment Station, University of Minnesota (1949).

(12) L. W. Gold, "Influence of Snow Cover on Heat Flow from the Ground," Association Internationale D'Hydrologie Scientifique Union Geodesique et Geophysique Internationale Assemblee Generale de Toronto, Tome IV, pp. 13–21 (1958).

(13) D. C. Pearce and L. W. Gold, "Observations of Ground Temperature and Heat Flow at Ottawa," *Journal of Geophysical Research*, Vol. 64, No. 9, Sept. 1959, pp. 1293–1298.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON NUCLEAR METHODS FOR MEASURING SOIL DENSITY AND MOISTURE

Newly developed nuclear methods for measuring moisture and density of soils were the subject of this Symposium on Nuclear Methods for Measuring Soil Density and Moisture. The papers presented described the apparatus in detail, presented data obtained on specific projects, and evaluated the methods by comparing results with those obtained by the conventional sand-cone and oven drying methods.

The symposium, sponsored by Subcommittee R-8 on Research on Special and Construction Control Tests of ASTM Committee D-18 on Soils for Engineering Purposes, was held on Monday, June 27, 1960, during the Fourth Session of the Sixty-third Annual Meeting of the Society.

Mr. H. F. Clemmer,[1] of the Government of the District of Columbia Department of Highways, served as symposium chairman. Mr. Bonner Coffman, Consulting Engineer on Soils, presided over the session which was held in the form of a panel discussion. Mr. Coffman presented digests of the papers, following which the individual authors answered questions pertaining to their papers. Mr. Abraham Schwebel, of the National Bureau of Standards, acted as panel consultant.

The papers included in the symposium are:

Introduction—Bonner Coffman
Design and Application of the Nuclear-Chicago d/M Gage—O. Kenton Neville and Theodore W. Van Zelst
Design and Calibration of a Neutron Moisture Meter—K. N. Burn
Application of Nuclear Soil Meters to Compaction Control for Airfield Pavement Construction—Paul F. Carlton
Experiences with Nuclear Moisture and Density Surface Probes on O'Hare Field Project—John P. Gnaedinger
Comparison of Nuclear and Sand-Cone Methods of Density and Moisture Determinations for Four New York State Soils—Sidney Mintzer
Evaluation of Nuclear Moisture-Density Testing Equipment—W. N. Carey, Jr., J. F. Shook, and J. F. Reynolds
Panel Discussion

These papers with discussion were issued as *ASTM Special Technical Publication No. 293* entitled "Symposium on Nuclear Methods for Measuring Soil Density and Moisture."

---

[1] Since deceased.

SUMMARY OF PROCEEDINGS OF THE SESSION ON ROAD AND PAVING MATERIALS

A knowledge of the characteristics and properties of bituminous mixtures as well as the bituminous material itself has provided a broad field of research in the study of such mixtures and materials as used in roads and pavements.

Committee D-4 on Road and Paving Materials organized this session, and the papers were presented at the Thirty-third Session of the Sixty-third Annual Meeting of the Society on June 30, 1960, in Atlantic City, N. J.

Mr. E. A. Whitehurst, University of Tennessee, acted as Symposium Chairman and presided over the session.

The papers included in the session are:

Introduction—E. A. Whitehurst

Stress Deformation Characteristics of Sand Bituminous Mixtures—Jake Carpenter

The Effect of Compaction Temperature on the Properties of Bituminous Concrete—Ralph W. Kiefer

A Method for Measuring the Air Permeabilities of Asphalt Concrete Pavements—W. H. Ellis and R. J. Schmidt

The Influence of Asphalt Composition on its Rheology—R. S. Winniford

Emulsified Asphalt Tests and Specifications—K. E. McConnaughay

Laboratory Tests and Recommended Specifications for Rapid Setting and Coarse Aggregate Mixing Cationic Asphalt (Bituminous)—E. W. Mertens, L. D. Coyne, and E. D. Rodgers

These papers with discussions were issued as *ASTM Special Technical Publication No. 294* entitled "Papers on Road and Paving Materials (Bituminous)—1960."

FLOW PROPERTIES OF BULK SOLIDS[†][*]

BY A. W. JENIKE,[1] P. J. ELSEY,[2] AND R. H. WOOLLEY[2]

SYNOPSIS

Principles developed for rigid-plastic solids exhibiting Coulomb's properties are adapted to the analysis of the flow of bulk solids, a term which describes a vast array of materials like ores, concentrates, coal, broken rock, salt, cocoa. A suitable yield function is proposed and a method and apparatus to measure the yield function are described.

LIST OF SYMBOLS

A = area of the horizontal cross-section of a shear cell, 0.0341 sq ft,
c = cohesion lb per sq ft,
EYL = effective yield locus,
F = flow-factor,
f_c = unconfined yield pressure, lb per sq ft,
IYL = instantaneous yield locus,
N = vertical force in a load cell during shear, lb,
S = shearing force in a load cell, lb,
TYL = time yield locus,
V = vertical force in a load cell during consolidation, lb,
V_1 = vertical force in a load cell $= \sigma_1 \times A$, lb,
YL = yield locus,
γ = shearing strain rate, per sec; density lb per cu ft,
δ = slope angle of the effective yield locus,
ϵ = compressive strain rate, per sec,
ϵ_1 = major compressive strain rate, per sec,
ϵ_2 = minor compressive strain rate, per sec,
$\bar{\epsilon}$ = compressive strain rate vector, per sec,
Φ = angle of friction in the stress field for $\sigma = \frac{1}{2}f_c(1\text{-}\sin \Phi)$,
ϕ = angle of friction in the stress field,
ϕ_v = angle of friction in the velocity field,
σ = pressure, lb per sq ft,
$\sigma_0 = (\sigma)_{\tau=0}$ = yield tension at zero shear stress, lb per sq ft,
σ_1 = major pressure, lb per sq ft,

[†] Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

[*] This work was done in the course of the "Flow of Bulk Solids Project" in an attempt to provide a quantitative basis for the design of bulk solids storage and handling plant. The project is sponsored by: Utah Engineering Experiment Station, University of Utah; Engineering Foundation, United Engineering Trustees; American Iron and Steel Institute; Kaiser Engineers, Division of Henry J. Kaiser Co.

[1] Director, Bulk Solids Flow Project, University of Utah, Salt Lake City, Utah.

[2] Assistant Director and Research Engineer, respectively, Engineering Experiment Station, University of Utah, Salt Lake City, Utah.

σ_2 = minor pressure, lb per sq ft,
σ_v = vertical pressure in a load cell during consolidation, lb per sq ft,
σ_n = vertical pressure in a load cell during failure, lb per sq ft,
τ = shear stress, lb per sq ft,
τ_v = shear stress in a load cell during consolidation, lb per sq ft, and
τ_n = shear stress in a load cell during failure, lb per sq ft.

BULK SOLIDS

Bulk solids exhibit elastic-plastic and viscous properties as well as a time effect similar to the jelling of Bingham materials and to the upper and lower yield points in mild steel. The present state of knowledge precludes a complete analysis of the flow of such solids. However, valuable information can be obtained from a simpler analysis based on the assumption that a bulk solid is sufficiently closely approximated by a modified rigid-plastic Coulomb solid (1).[3] In this approach, velocity terms are neglected. In the practice of bulk solids flow, velocities are often low and the velocity terms are often insignificant. In all problems of incipient flow, velocities are in fact equal to zero.

The concept of the modified rigid-plastic Coulomb solid can be described as follows: a rigid-plastic solid is one for which there exists a limiting stress function such that stresses lower than those determined by the function cause no deformation, whereas stresses equal to the limiting stresses cause either failure or plastic flow. Stress conditions exceeding the limiting stresses are not permissible.

Calling the shearing stress τ, the normal pressure σ, and using the (σ,τ) system of coordinates, the limiting stress function is represented by a yield locus, *YL*. For a Coulomb solid, the yield locus is a straight line, inclined at an angle of friction ϕ to the σ-axis and intersecting the τ-axis at a value of τ equal to c, which is called the cohesion of the solid, Fig. 1. In the (σ,τ) system of coordinates any stress condition can be represented by a Mohr semicircle. A rigid-plastic Coulomb solid remains rigid for stresses represented by a Mohr semicircle *A*, which lies below the yield locus, and deforms for stresses determined by a Mohr semicircle *A′* tangential to the locus. Stresses corresponding to a Mohr semicircle intersecting the yield locus are not permissible.

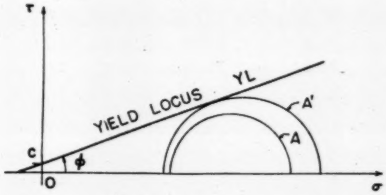


FIG. 1.—Yield Locus of a Coulomb Solid.

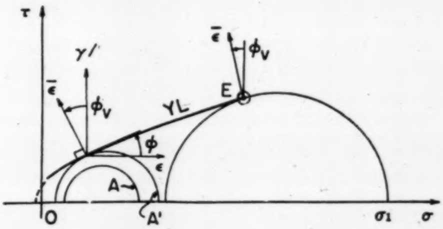


FIG. 2.—Yield Locus of a Bulk Solid.

On the basis of measurements of the yield locus of bulk solids, we conclude that the Coulomb concept has to be modified in the following ways:

1. For small values of normal pressure σ, the locus deviates significantly from a straight line. The probable shape of the yield locus, *YL*, is shown in Fig. 2.

2. The yield locus does not extend

---

[3] The boldface numbers in parentheses refer to the list of references appended to this paper.

indefinitely with increasing σ but terminates at a point E.

3. The position of the yield locus is not constant but is a function of the degree of consolidation of the solid. An unconsolidated, loose bulk solid has no strength at all and its yield locus reduces to a point at the origin 0. As consolidation (and density) increases, the yield locus expands.

It seems necessary to assume that, in general, the shape of the yield locus of a given bulk solid is a function not only of its density but also of the relative positions of the particles, and that the method of consolidation may affect the shape and position of the locus. In order to circumvent this difficulty a method of consolidation has been adopted in this work which, on the one hand, closely reproduces the consolidation that a solid undergoes while flowing in a channel and, on the other hand, is readily produced with our testing apparatus. This method so proportions the consolidating pressures that material always shears as it compacts, and the degree of consolidation is fully described by one value, say, that of the major consolidating pressure σ_1.

A yield locus determines not only the stress conditions for which flow occurs but also the direction of the strain rate vector $\bar{\epsilon}$ during flow. In Fig. 2 let us identify the directions of the axes σ and τ with the directions of ϵ and γ, the compressive and shearing strain rates, respectively. Then by the principle of normality (2) we can say that, for any limiting stress condition determined by a Mohr semicircle A', the direction of the strain rate vector $\bar{\epsilon}$ is normal to the yield locus at the point of tangency. The direction of vector $\bar{\epsilon}$ is measured by angle ϕ_v which the vector forms with the γ-axis. For negative values of ϕ_v the strain rate vector has a positive compressive strain rate component ϵ. This implies consolidation and an increase of yield strength signified by an expansion of the yield locus. Contrariwise, positive values of angle ϕ_v imply expansion of the solid which is opposite to consolidation and, therefore, a reduction in density and a shrinking of the yield locus. For a stress condition corresponding to a Mohr semicircle tangential to the yield locus at the terminus E, the principle of normality does not provide a unique direction for $\bar{\epsilon}$ but restricts angle ϕ_v to a range: -90 deg $< \phi_v \leqslant \phi$.

The meaning of the yield locus can now be stated as follows: The yield locus, Fig. 2, has been generated by a consolidation with shear measured by the major pressure σ_1. The Mohr semicircle through σ_1 and tangential to the yield locus determines the terminus E of the locus. The bulk solid is rigid for any stress condition represented by a Mohr semicircle A not touching the yield locus. When the stress condition A changes to A' so as to touch the yield locus the solid shears. When the solid shears, the direction of the strain rate vector $\bar{\epsilon}$ is normal to the yield locus, ϕ_v is equal to ϕ and is positive, the solid expands, density decreases, the yield locus shrinks rapidly until it has assumed the shape corresponding to the new pressures, which establish a new degree of consolidation. The stress condition determined by the Mohr semicircle A' is of brief duration and there is a jump from one yield locus down to another locus at a lower strength level.

The jump is greatest when the Mohr semicircle A approaches the yield locus for low values of σ. As the semicircle approaches the yield locus closer and closer to the terminus E, the magnitude of the jump diminishes, until at E there is no jump at all but a condition of continuous plastic flow. During continuous flow the pressures may remain constant, the solid then flows in pure shear with no change in density and no change of the yield locus; the direction of the strain rate vector $\bar{\epsilon}$ is parallel to the τ-axis, $\phi_v = 0$. When the pressures increase continuously consolidation progresses, the density of the solid increases, the yield locus expands while the angle ϕ_v of the strain rate vector is negative. When the pressures decrease the reverse takes place.

A bulk solid may also fail in tension. This is often referred to as brittle failure. In the yield diagram this is represented by a stress semicircle approaching the yield locus at $\tau = 0$. The tension side of the yield locus is shown to a larger scale in Fig. 3. The boundary between tension failure and shear failure is defined by a limiting semicircle which has the curvature $\frac{1}{r}$ of the yield locus at point, σ_0,0. All stress conditions represented by semicircles contained within the limiting semicircle approach the yield locus at point, σ_0,0, and cause tension failure. It should be noted that for $\tau = 0$, the angle of friction ϕ of the yield locus must be equal to 90 deg in order that r be greater than zero, and that tension failure might occur for a stress other than hydrostatic tension σ_0. In addition, if angle ϕ were less than 90 deg for $\tau = 0$, as shown in Fig. 4, then the solid would be stable for a hydrostatic tension σ_h but would fail if the tensions were reduced to the values shown by the Mohr semicircle. This is contrary to experience. Thus, it would seem that ϕ is equal to 90 deg for $\tau = 0$ for all yield loci whose intercept with the τ-axis is above zero.

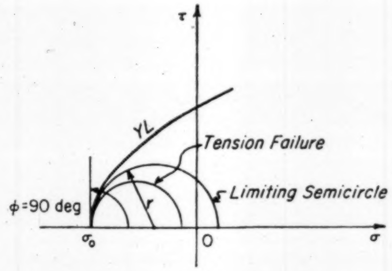


FIG. 3.—Failure in Tension.

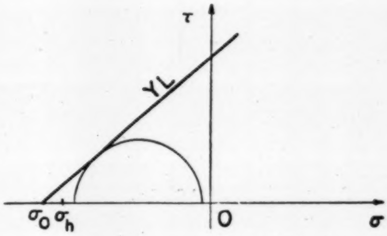


FIG. 4.—Unlikely Shape of the Yield Locus.

A bulk solid may also undergo consolidation without shear. This occurs when the mean pressure is sufficiently high while the deviator components are insufficient to cause shear. This method of consolidation is of relatively lesser importance in the flow of bulk solids and, therefore, it has not been studied except in so far as it occurs in the "Time Effect" which is discussed below.

Continuous Flow.—Under conditions of continuous flow, an element of the flowing solid continuously reconsolidates under the prevailing pressures. The yield loci and flow properties measured to represent these conditions will be called "instantaneous yield loci," *IYL*, and "instantaneous flow properties." When necessary to avoid confusion these flow properties will be defined by a subscript *i*.

Time Effect.—When the flow of a bulk solid in a channel is stopped the solid remains under the action of the prevailing static pressures. Experience shows that under these conditions a solid usually gains strength with the lapse of time. When flow is later about to be restored, the incipient pressures necessary to overcome the newly acquired yield strength are higher than those prevailing during continuous flow. If these higher pressures fail to develop, flow does not occur. The yield loci and flow properties obtained for a solid that has consolidated for a period of time will be called "time yield loci," *TYL*, and "time flow properties." These flow properties will be identified by a subscript *t*. The degree to which the time yield locus increases above the continuous flow or instantaneous yield locus depends on the solid. There are many materials which are quite free flowing under conditions of continuous flow but which become nonflowing when allowed to consolidate at rest.

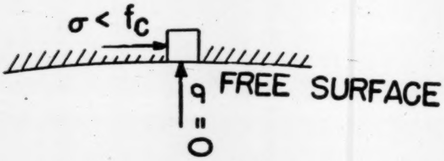


FIG. 5.—Pressures at a Free Surface.

The time effect is particularly pronounced when the particles of the solid are soft, or are softened by raising the temperature of the stored solid. The time effect is also significant when the solid contains a water solution of salts, and water is allowed to evaporate during storage at rest. This precipitates the salts which cement the particles.

Flow Properties:

Flow-function (3).—At a free surface of a mass of solid, Fig. 5, the minor pressure is normal to the surface and is equal to zero. The largest pressure which the solid can support in the direction tangential to the surface is, therefore, a major pressure, call it f_c, which corresponds to zero minor pressure. f_c is often referred to as the unconfined yield pressure. When the yield locus of a bulk solid is known, f_c is found by drawing a Mohr semicircle tangentially to the locus and through the origin, Fig. 6. In order that a bulk solid be capable of forming a free surface inclined to the horizontal at an angle greater than the angle of internal friction ϕ the solid must have a value of f_c greater than zero (4, 5). Thus a perfectly free-flowing solid is one for which $f_c = 0$ for all degrees of consolidation. During continuous flow with shear, to each value of the major consolidating pressure σ_1, there corresponds a unique yield locus which determines a value of f_c. The function

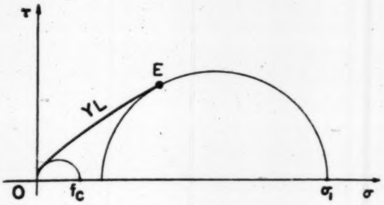


FIG. 6.—Determination of f_c.

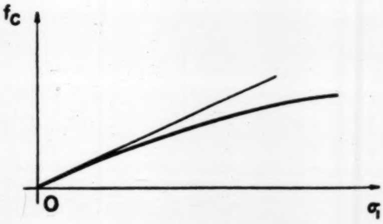


FIG. 7.—Relation $f_c = f(\sigma_1)$.

$$f_c = f(\sigma_1) \qquad (1)$$

is shown in Fig. 7 and expresses the most important flow property of a bulk solid. It is called the flow-function. A large number of tests have shown that, in the useful range of pressure σ_1, the flow-function curve is convex. The degree of curvature is often small and this relation is then sufficiently well represented by a straight line tangential to the curve at the origin. This relation is called the flow-factor[4] and is defined as:

$$F = \left(\frac{d\sigma_1}{df_c}\right)_{\sigma_1 \to 0} \quad \ldots\ldots\ldots\ldots (2)$$

Thus for $F = \infty$, $f_c = 0$ and the solid is perfectly free flowing. For $F < 1$, the unconfined yield pressure f_c is greater than the major consolidating pressure σ_1. Such a solid will not flow by gravity alone.

The effect of time on the value of f_c is illustrated in Fig. 8. The time yield locus, *TYL*, lies above the instantaneous yield locus, *IYL*, and the corresponding value of $(f_c)_t$ is considerably larger than $(f_c)_i$ for the same major consolidating pressure σ_1, therefore, the time flow-factor will be correspondingly smaller than the instantaneous flow-factor.

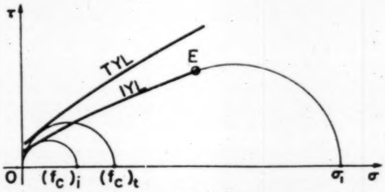


FIG. 8.—Time Effect.

Effective Yield Locus (1).—During continuous flow with shear, to each major pressure σ_1 there corresponds a unique minor pressure and, therefore, a unique Mohr semicircle. The effective yield locus is defined as the envelope of these Mohr semicircles as σ_1 varies. The construction of the effective yield locus, *EYL*, is shown in Fig. 9. This property is required in the mathematical analysis of stress fields occurring during the continuous flow of bulk solids. In general, the effective yield locus is a curved line which passes through the origin. It is quite likely that a straight line through the origin can approximate the locus sufficiently well, in which case an approximate effective yield locus can be defined by an angle δ as shown in the figure.

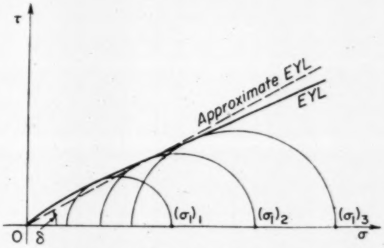


FIG. 9.—Effective Yield Locus.

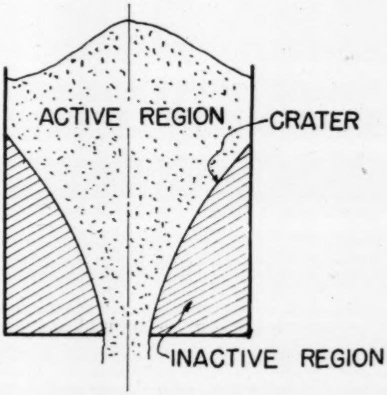


FIG. 10.—A Flow Channel.

When a solid is placed in a channel, as shown in Fig. 10, some material on the outside remains inactive while flow occurs only within the crater. Angle δ has a bearing on the slope of the crater. Solids possessing a large angle δ form steeper craters, and *vice versa*. As a result, angle δ affects the slope of the stable bank that a solid can form, because material in an inactive region tends to consolidate with time and to remain stable even after the crater has emptied out. Thus, the larger δ is, the steeper the stable slope that material can form.

---

[4] In Jenike's previous publications relation $\frac{f_c}{\gamma} = f\left(\frac{\sigma_1}{\gamma}\right)$ was called the flow-factor of a bulk solid.

Susceptibility to Vibration.—The yield loci of bulk solids show that the tensile strength of these materials is quite small. Nonetheless, steep craters and domes can form in these materials because in these structures the weight of the material can be transferred by means of compressive stresses. However, when vibrations are applied to a mass of material, momentary tensile stresses arise and, if the tensile strength of the solid is insufficient, steep slopes and domes collapse. Thus, a measure of the susceptibility to vibration is provided by the function

$$\sigma_0 = F(\sigma_1) \ldots\ldots\ldots\ldots (3)$$

where σ_0 is the largest yield tension, that is the yield tension for zero shear stress.

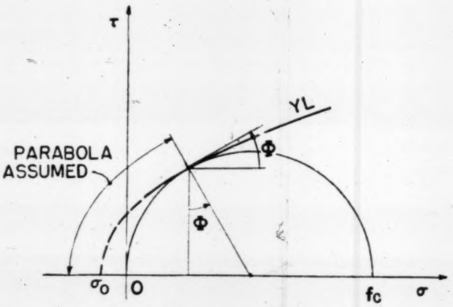


FIG. 11.—Determination of σ_0.

While we have no way of measuring σ_0 directly, its value can be estimated if we make an assumption concerning the shape of the yield locus. For instance, let us assume that in the vicinity of σ_0 the locus is in the shape of a parabola, which is symmetric with respect to the σ-axis and tangential to the yield locus at the point of contact with the Mohr semicircle by which f_c was determined, Fig. 11. Then the following relation is obtained:

$$\sigma_0 = f_c \frac{(1 - \sin \Phi)^2}{4 \sin \Phi} \ldots\ldots\ldots (4)$$

where Φ is the angle of friction measured at the point of contact.

Using the flow-function, Eq 1,

$$\sigma_0 = f(\sigma_1) \frac{(1 - \sin \Phi)^2}{4 \sin \Phi} \ldots\ldots\ldots (5)$$

is obtained. Evidently, the steeper the slope of the yield locus at the point of contact, the smaller is the tensile yield stress σ_0, and the less stable are the obstructions to flow.

TESTING

Apparatus:

Early work (3) showed evidence of a similarity between the behavior of bulk solids and soil media. Testing apparatus developed in soil mechanics was therefore applied to the measurement of the cohesion and of the angle of internal friction of bulk solids. Standard direct shear apparatus and unconfined compression apparatus were tried. The results obtained with shear apparatus were not satisfactory primarily because the range of values of cohesion for which the soil test machines were designed was outside the useful range for bulk solids. By the terminology of soil mechanics, bulk solids can be considered cohesionless and cohesive soils under their usual degree of consolidation would not flow by gravity in any storage system. Unconfined compression apparatus was found unsuitable because of the impossibility of applying a known, uniform degree of consolidation and because the stresses due to the weight of the sample were in the range of the measured yield stresses. These samples often disintegrated during handling. The application of a triaxial compression apparatus was considered and rejected as impractical because of the considerable time required to obtain results by this method. It was decided to develop a direct shear test machine especially for testing bulk solids.

A direct shear test machine was designed and build in 1953. It has since undergone many refinements. A machine now in use at the Bulk Solids Flow Laboratory is shown in Fig. 12. It is equipped with a shear cell, a gravity vertical loading system, and an electric-pneumatic shearing force device. The shearing force device has a constant strain-rate of 0.036 in. per min. The shearing force necessary to maintain the strain-rate is continuously recorded on a pneumatic recorder. This arrangement produces a permanent record of the stress-strain relations for each test. A test cell is shown in Fig. 13. It is composed of a base located on the frame of the machine, a ring resting on top of the base, and a cover. The bottom of the cover and the inside of the base are roughened to increase adhesion of the tested solid.

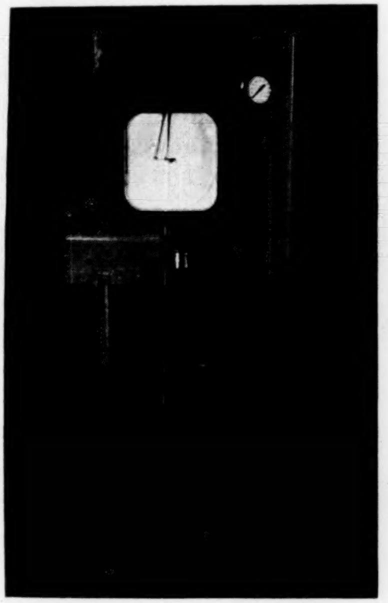


FIG. 12.—Direct Shear Apparatus.

The base and the ring are filled with the tested solid. The vertical compressive force *V* is applied to the cover. The horizontal shearing force *S* is applied by means of a stem which acts on a bracket attached to the cover. The stem acts in the plane of contact between the ring and the base. A part of the shearing force is transferred from the bracket to the ring through a loading pin. The difference between the inside diameter of the ring and the outside diameter of the cover is 0.04 in. The machine is capable of applying a vertical force up to 100 lb and a shearing force up to 60 lb.

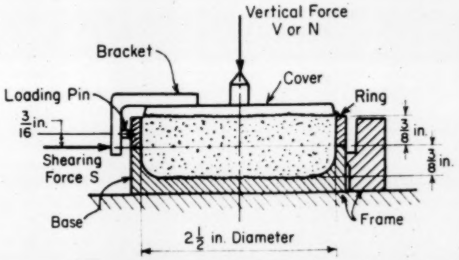


FIG. 13.—Shear Cell.

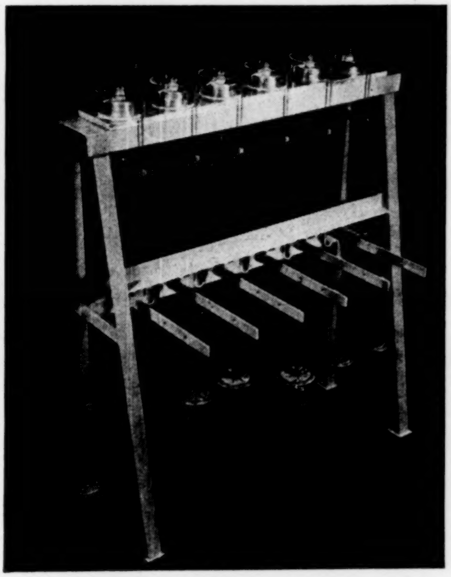


FIG. 14.—Consolidating Bench.

The shear test machine is used in conjunction with a six cell consolidating bench in which the whole loading cells are placed for time consolidation, Fig. 14.

Procedure:

The purpose of the tests is to measure the flow properties of bulk solids for two consolidating conditions: first, for the condition of continuous flow, second, for the time effect of consolidation at rest after flow has stopped and material is left under pressure.

The tests give significant results provided the procedure is such as to assure: (*a*) a representative specimen, (*b*) a uniform specimen, and provided that the stresses occurring during (*c*) consolidation, and (*d*) shear are known with sufficient accuracy. While these requirements sound commonplace, they are by no means easy to satisfy. Both the apparatus and the procedure described herein are the result of extensive experimentation. It is believed that the described method has brought the errors of tests within the range of scatter inherent in bulk solids flowing in a channel.

Continuous Flow:

(*a*) *Representative Specimen.*—In general, the yield locus of a material for a given consolidating stress and given strain-rate is affected by the water content, temperature, and particle size distribution of the specimen. The tests have been conducted on solids containing water below the point of saturation. In view of the considerable influence that water content has on the yield loci of many materials it is necessary to reproduce the required content in the specimen accurately and to handle the specimen rapidly so as to minimize errors through evaporation.

When temperature is a significant factor, the solid and the consolidating bench are placed in a heated chamber. The tests are carried out when the solid has reached the required temperature.

The particle size distribution is not as perplexing as it might appear at first sight. In the flow of bulk solids, our interest lies in the conditions that lead to stoppages of flow which we want to avoid. Stoppages occur when the solid reaches a sufficiently high yield strength. Thus, the critical conditions to be considered are those of highest strength. During the flow of a mass of mixed particle sizes, the large particles move bodily while the material shears across the fines. Therefore, the yield strength of the mass depends on the properties of the fines. The coarse particles are a passive agent and like aggregate in concrete they do not develop yield strength without fines to bind them. Experience shows that screened out coarse ores in bulk are invariably free flowing; they do not develop any yield strength. To determine the yield loci of a mixed material the fines are screened out through an arbitrarily chosen No. 20 mesh (0.033 in. aperture) sieve and tested.

(*b*) *Uniform Specimen.*—The consolidation of the sample is carried out in two stages. The purpose of the first stage is the preconsolidation of a uniform specimen. With the cover off the test cell, a packing mold is placed on top of the ring and both the mold and the ring are placed in an offset position on the base, as shown in Fig. 15. A specimen of the tested solid is then placed in the cell. One layer after another is slightly packed with the fingers, up to the top of the mold. The excess material is scraped off level with the top of the mold. A twisting top is placed over the specimen. The top is essentially like the cover of Fig. 13 but with a smooth bottom surface and without the bracket. A vertical force V is applied to the top by means of the system of lever and weight shown in Fig. 12. Force V causes a vertical pressure σ_v in the material. By means of a special wrench, an oscillating twist is now applied to the cover. It has been found by experience that between twenty and sixty reversals of the twisting motion are necessary to obtain a uniformly packed sample. The full angle of twist is about 10 deg. This preconsolidates the solid and assures a uniform specimen.

(*c*) *Consolidation.*—Consolidation is completed by causing the specimen to flow with shear under given stresses until a steady state is closely approached. This is attained in the following way: Load *V* is released, the twisting top and the mold are removed, the excess material is scraped off level with the top of the ring and the test cover is placed on the material in a position offset 0.02 in. in the direction opposite to the shear strain, thus leaving a gap of about 0.02 in. between the loading pin and the ring. Load *V* is now replaced and a shearing force *S* is applied to the bracket, Fig. 13, by means of the electric-pneumatic shearing device. As the strain proceeds, the cover slips or flows over the top of the specimen, distributing the applied shearing force over the top area of the specimen. The slipping (or flow) of the cover stops when the loading pin comes in contact with the ring, and now a part of the shearing force is also applied through the ring. This method of loading seems to assure a fairly uniform distribution of the shearing stresses across the specimen. Prior to the adoption of this method, the shearing force used to be applied directly to the ring. This used to cause a distinct stress concentration at the loaded edges of the ring and of the base. Material in the ring would pack against the loaded half of the ring, causing an empty gap to appear along the unloaded half of the ring. Furthermore, the unloaded side of the ring would often rise off the base, exposing the sheared specimen and weakening it significantly. It will be noted from Fig. 13 that the loading pin is raised $\frac{3}{16}$ in. above the plane of shear and, as a result, the tendency of the ring to rise is distinctly reduced. As an additional safeguard against the rising of the ring, it is sometimes pressed down with the fingers at intervals during the shearing motion.

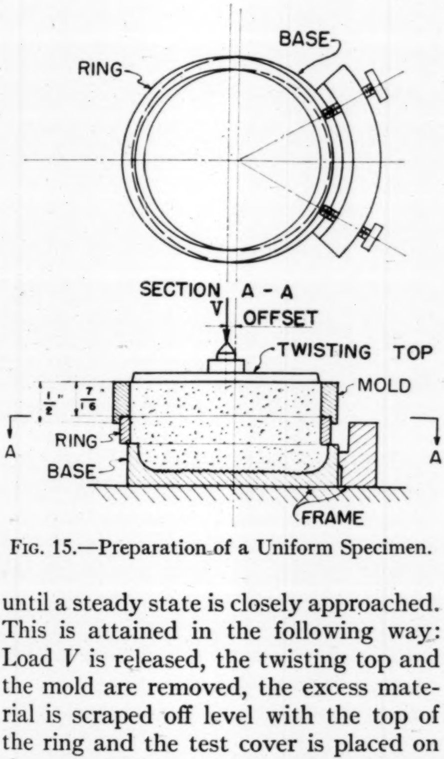
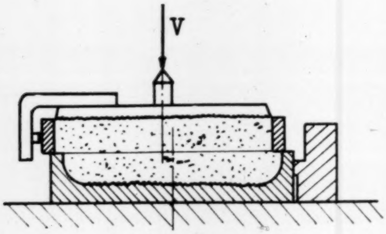
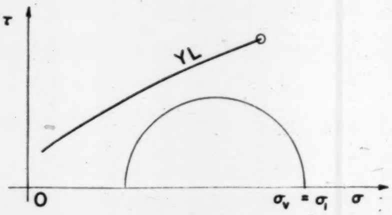


FIG. 15.—Preparation of a Uniform Specimen.

As the strain proceeds, a condition is reached when a layer of material across the whole specimen is caused to flow plastically; the recorded shearing force reaches a steady value. The stresses which occur in the specimen during this stage of consolidation can be visualized as follows: the process starts with only the vertical pressure $\sigma_v = V/A$, where A is the area of the horizontal cross-section of the specimen (Fig. 16(*a*)), σ_v is then the major stress and the corresponding Mohr semicircle is shown in Fig. 16(*b*) together with some yield locus which was developed during the twists of preconsolidation. The Mohr semicircle is below the yield locus and no flow occurs at this stage. Now the shearing stress $\tau_v = S/A$ is applied. As τ_v increases, the direction of the major stress σ_1 rotates away from the vertical until a steady state is reached for which the direction of



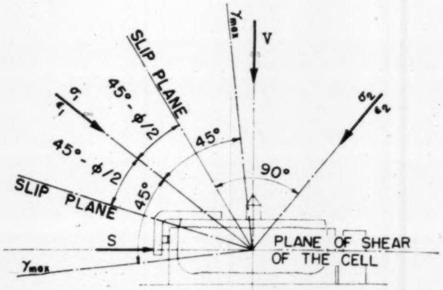
(*a*) Loading



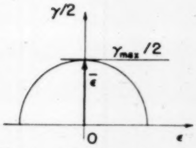
(*b*) Stresses.

FIG. 16.—Beginning of Consolidation with Shear.

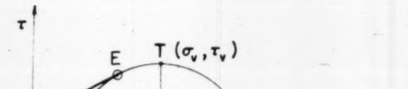
σ_1 is shown in Fig. 17(*a*). For a steady state, there is no change of density, the strain rate vector $\bar{\epsilon}$ acts in the direction of axis $\gamma/2$, and the center of the Mohr semicircle of strain rates, Fig. 17(*b*), is located at the origin of the system of coordinates $\left(\epsilon, \dfrac{\gamma}{2}\right)$. For this condition the compressive strain-rate, normal to the plane of greatest shear (plane ($\gamma_{\max}$), is equal to zero. If the specimen were perfectly restricted in the cell, the height of the specimen would remain constant and the plane of greatest shear ($\gamma_{\max}$) would be forced to coincide with the plane of shear of the cell. The principal strain rates ϵ_1 and ϵ_2 which are inclined at 45 deg to the plane of greatest shear would be inclined at the same angle to the plane of shear of the cell. Assuming isotropy, the directions of the principal pressures σ_1 and σ_2 coincide with the directions of the principal strain-rates ϵ_1 and ϵ_2 and would also be inclined at 45 deg to the plane of shear of the cell. The vertical pressure σ_v would then be inclined at


V
γ_max
SLIP PLANE
σ_1 ε_1
45° - φ/2
45°
45° - φ/2
45°
90°
σ_2 ε_2
SLIP PLANE
S
PLANE OF SHEAR OF THE CELL
γ_max

(*a*) Loading.


γ/2
γ_max/2
ε̄
O
ε

(*b*) Strain Rate Mohr Circle.


τ
E
T (σ_v, τ_v)
O
σ_v
σ_1
σ
c
τ
E
(σ_v, τ_v)
O
σ_v
σ_1
σ
d
τ
ε̄
φ_v = φ
E
R
T
YL
(σ_v, τ_v)
(σ_n, τ_n)
O
σ_n
σ_2
σ_v
σ_1
σ
e

(*c*), (*d*), (*e*) Possible Stress Conditions.

FIG. 17.—Steady State Flow During Consolidation With Shear.

45 deg to the principal pressures. The measured stresses (σ_v, τ_v) would determine point T at the top of the Mohr semicircle, Fig. 17(*c*), (7).

On the other hand, if the specimen were quite unrestricted, one can speculate that one of the slip planes of the specimen would coincide with the plane of shear of the cell. The major pressure σ_1 is inclined at an angle 45 deg $- \phi/2$ to the slip planes. This would locate the direction of the major pressure σ_1 at 45 deg $+ \phi/2$ from the vertical pressure σ_v and the recorded shearing stress τ_v would now lie at point E, the terminus of the yield locus, Fig. 17(*d*).

In fact, the specimen is partially restricted and the recorded point R falls on the Mohr semicircle between E and T, Fig. 17(*e*). The degree of restriction varies within certain limits between one test and another; therefore, the position of point R on the Mohr semicircle also varies somewhat. The determination of the yield locus requires the measurement of at least two and usually more points (σ_n, τ_n) on the locus. For each point, the specimen is first consolidated and then sheared. It is necessary to reproduce the same degree of consolidation each time. To attain this goal, the shearing strain during consolidation is not continued to the steady state but is interrupted when the shearing force reaches a given value chosen at about 95 per cent of the expected steady value. (A preliminary test is run first to determine the approximate steady value of the shearing force.) This assures an acceptable scatter of the (σ_n, τ_n) points. The amount of the offset of the ring over the base is so adjusted that by the time this desired degree of consolidation is reached the ring is in a central position.

(*d*) *Shear.*—When consolidation is completed, the stem of the shearing force device is retracted. The vertical compacting load V is replaced by a smaller load N, such that pressure $\sigma_n = N/A$, Fig. 17(*e*), will locate a useful point of the yield locus. The shearing force is now applied until a failure plane has developed. That fact is indicated on the recorder by the force passing a maximum value. After shearing, the plane of failure of the specimen is checked. It should coincide with the plane of shear of the cell as shown in Fig. 18. If the planes do not coincide it means that the measured point (σ_n, τ_n) does not lie on the yield locus and the test is repeated. Tests are begun with the lowest required normal pressure σ_n. After each successful test, pressure σ_n is stepped up until a sufficient number of points is obtained to determine the yield locus. Experience shows that the largest value of σ_n for which a coincidence of the plane of failure with the plane of shear of the cell can be expected equals about $\frac{3}{4}$ of σ_v. When the yield locus has been determined, the major consolidating pressure σ_1 is found by extrapolating the locus toward the higher values of σ and by drawing a Mohr semicircle through point (σ_v, τ_v) tangentially to the yield locus. The points of intersection of the semicircle with the σ-axis determine the values of the principal pressures σ_1 and σ_2.

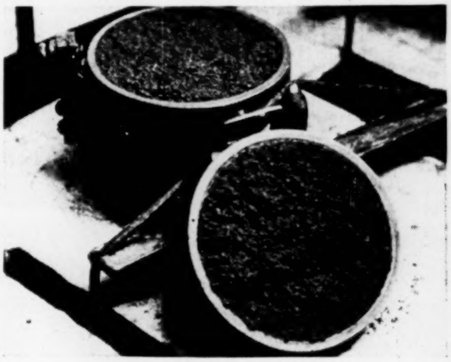


FIG. 18.—Plane of Failure of a Specimen.

Example.—A recorder chart of the tests of a lead concentrate is shown in Fig. 19. The curves are numbered and represent the following steps:

1. Preliminary test to determine the value of the steady flow shearing stress corresponding to a vertical pressure σ_v.

2. Consolidation of a test specimen under the same pressure σ_v and a shearing stress τ_v equal to about 95 per cent of the steady value. The jogs in the recorded lines occurred when the ring was pressed down with the fingers.

3. The consolidated sample is sheared under a normal pressure σ_n, giving a shearing stress τ_n at failure.

4. Same as (2).

5. Same as (3) but σ_n is stepped up and a higher value of τ_n is obtained.

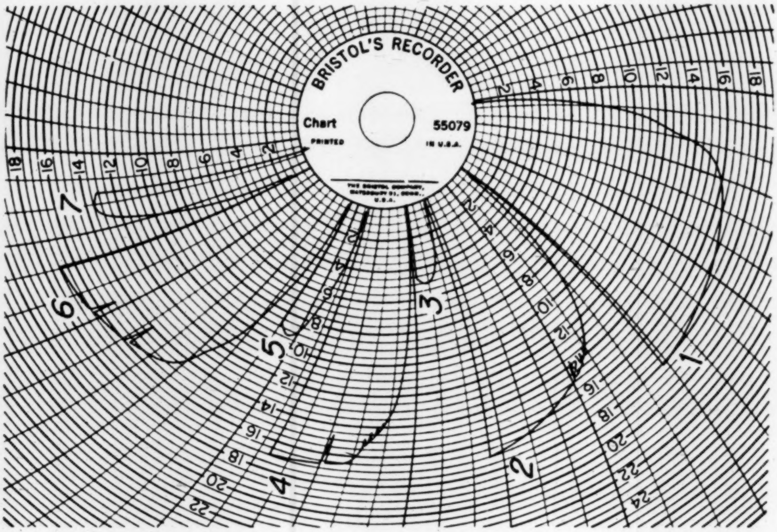


FIG. 19.—Typical Recorder Chart.

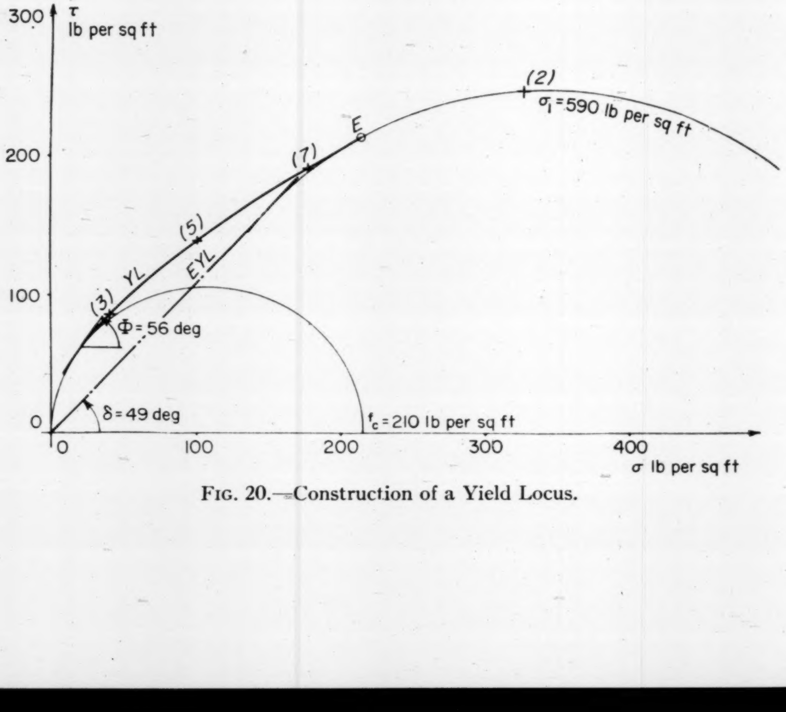


FIG. 20.—Construction of a Yield Locus.

6. Same as (2).

7. Same as (5) but σ_n is again stepped up and a higher value of τ_n is obtained.

The values obtained are as follows:

	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Test 7
V, lb.....	11.1	11.1	...	11.1	...	11.1	...
σ_v, lb per sq ft....	326	326	...	326	...	326	...
N, lb......	...	...	1.1	...	3.1	...	6.1
S_n, lb per sq ft....	...	...	32	...	91	...	179
Chart reading......	18.1	17.0	5.1	17.0	9.3	17.0	13.4
S, lb......	9.1	8.6	2.7	8.6	4.7	8.6	6.8
τ_v, lb per sq ft....	267	252	...	252	...	252	...
τ_n, lb per sq ft....	...	...	79	...	138	...	199

Points (σ, τ) obtained in tests 2, 3, 5 and 7 are plotted in Fig. 20. A smooth curve is drawn through points 3, 5 and 7, and extended toward point 2. A Mohr semicircle is drawn through point 2 tangentially to the curve. The point of tangency determines the terminus E of the yield locus and the point of intersection of the circle with axis σ (outside the figure) defines the major consolidating pressure $\sigma_1 = 590$ lb per sq ft. Another Mohr semicircle is drawn through the origin and tangentially to the yield locus. The point of intersection of this circle with axis σ determines the value of the unconfined yield pressure $f_c = 210$ lb per sq ft. The first approximation of the flow-factor is obtained from the formula

$$F = \frac{\sigma_1}{f_c} = \frac{590}{210} = 2.8$$

The first approximation of the effective yield locus angle is obtained by drawing a straight line through the origin, tangentially to the Mohr semicircle which passes through point E; $\delta = 49$ deg.

Time effect.—To reproduce the time effect in the tests, the specimen is first prepared and consolidated in the way described for continuous flow and then is left to consolidate on the consolidating bench under a static stress condition. It is assumed that the stresses in a flow channel remain essentially the same after flow has stopped as they were during continuous flow. To reproduce this static condition in the specimen, a vertical force $V_1 = A \times \sigma_1$ is applied, where σ_1 is the major consolidating pressure obtained from the yield locus for continuous flow. No shearing force is applied to the cell. Load V_1 is left on the specimen for the prescribed period of time after which the specimen is sheared in the manner described for continuous flow. The yield loci are plotted and the flow-factor is obtained in the usual way.

During the static compaction it is necessary to preserve the moisture content of the specimen. This is done by covering the shear cells with plastic cups shown in Fig. 14; the bases of the cups are sealed by baths of mercury.

REFERENCES

(1) A. W. Jenike and R. T. Shield, "On the Plastic Flow of Coulomb Solids Beyond Original Failure," *Journal of Applied Mechanics*, Dec. 1959, pp. 599–602.

(2) D. C. Drucker, "A More Fundamental Approach to Plastic Stress-Strain Relations," *Proceedings*, First U. S. National Congress of Applied Mechanics, Am. Soc. Mechanical Engrs., pp. 487–491 (1951).

(3) A. W. Jenike, "Flow of Bulk Solids," *Bulletin No. 64*, University of Utah, Utah Engineering Experiment Station, March 1954.

(4) D. W. Taylor, "Fundamentals of Soil Mechanics," John Wiley & Sons, Inc., New York, N. Y. (1948).

(5) V. V. Sokolovski, "Statics of Earthy Media," State Publishing House of Technical and Theoretical Literature, Moscow (1954).

(6) A. W. Jenike, P. J. Elsey, and R. H. Woolley, "Flow of Bulk Solids," Progress report, *Bulletin No. 96*, University of Utah, Utah Engineering Experiment Station, Dec. 1958.

(7) R. Hill, "The Mathematical Theory of Plasticity," p. 295, Oxford Press (1950).

DISCUSSION

Mr. H. Colijn[1] (*presented in written form*).—I would like to compliment the authors on their interesting paper. It is gratifying to note that after about 50 years of relative standstill the subject of bulk solids flow has again attracted the attention of engineers and scientists.

The design of storage bins, hoppers, and connecting spouts has been based generally on previous experience. The many battered hoppers to be seen in bulk material handling systems are mute evidence of the inadequacy of this approach to design. It is disconcerting to find that a modern factory equipped with the latest automatic controls must depend in varying degrees upon manual poke holes or rapping devices to assist flow of bulk solids. The extrapolation of inadequate empirical data in this field cannot assure safe limits in the design of bins and hoppers. Nor can precautionary overdesign, with its additional capital investment of uncertain need, be considered an acceptable substitute for sound engineering procedure. Such sound procedure can only be founded on clear understanding of the behavior of materials during storage and flow.

Excellent beginnings were made in the scientific analysis in this field at the turn of the century. Names such as H. A. Janssen, Wilfred Airy, W. J. M. Rankine are closely related to the studies of pressures and stresses in granular materials.

It has been greatly through the efforts of Mr. Jenike that new interest has been created in the scientific approach of flow of bulk solids. His experimental work and endeavors to work out a theory of gravity flow have stimulated others to participate and study the problems involved.

As a result of the present interest in bulk solids flow The American Society of Mechanical Engineers, for instance, has established a research committee on flow of bulk materials, which has worked out an extensive research program both of a theoretical and experimental nature.

In discussing the present paper I would like to make the following comments:

First of all, I would like to emphasize the testing procedures which the authors have developed. They deviate considerably from the standard shear testing procedures used in soil mechanics. This is mainly the result of the very small cohesion values which have to be measured. Variations in cohesion of the order of magnitude of 5 lb per sq ft are important when we consider gravity flow in hoppers. A variation of 1 per cent moisture in the aggregate can mean flow or no flow in a particular hopper.

There are various shear testing methods presently under evaluation at our Research Center to determine which one gives the most accurate and consistent test results. We do not intend to tie the testing program as closely to gravity flow as the authors did. Our purpose is to measure the shear strength properties of the granular bulk solid so that these properties can be used in the analysis in various fields of bulk solid handling, which may include, besides gravity flow, vehicle performance on a stock pile, behavior of bulk solids in transport carriers such as ships and railroad cars, (self-cleaning buckets) in bucket elevators, on belt conveyors, etc.

---

[1] Applied Research Laboratory, U. S. Steel Corp., Monroeville, Pa.

The authors consider the yield locus of a bulk solid to be a function of the degree of consolidation, and they consider the degree of consolidation to be represented by the "consolidation pressure." Since the degree of consolidation can also be represented by bulk density, we wonder why it is necessary to introduce "consolidation pressure" at all. Bulk density is a clearly defined and measurable material property, whereas consolidation pressure is not so clearly defined. The authors imply, for instance, that consolidation pressure with shear has a different effect than consolidation pressure without shear. Why not measure bulk density rather than consolidation pressure? This, in fact, seems to be the approach adopted by many workers in soil mechanics.

Another point that seems to need clarification concerns the curvature of the yield locus. In particular, the authors conclude that the yield locus should meet the σ-axis at 90 deg in the σ-τ diagram. This conclusion, they say, is based upon experience. Mr. Richmond of The United States Steel Corp. Research Center suggests the possibility that there are two mechanisms of failure in bulk materials just as there are in metals, one for yielding and one for fracture. The σ-τ diagram for an ideal plastic solid can be represented, for instance, by the accompanying Fig. 21. Is it not possible that the diagram for a bulk material is as illustrated in Fig. 22?

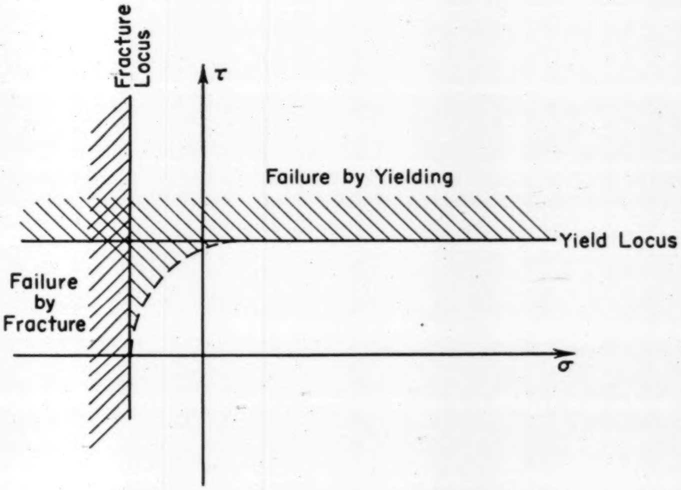


FIG. 21.—σ-τ Diagram for an Ideal Plastic Solid.

The theory of stress-strain relationship discussed in the first part of this paper is somewhat speculative. A more detailed discussion of this theory is given in the Shield-Jenike paper published in the December 1959 issue of *Journal of Applied Mechanics*.[2] As far as I know there have been no real problems worked out using this theory. The theory depends heavily on the assumption that the principle of normality applies during the plastic deformation of bulk solids or in other words, that the direction of the strain vector is perpendicular to the yield locus or yield surface.

---

[2] R. T. Shield and A. W. Jenike, "On the Plastic Flow of Coulomb Solids Beyond Original Failure," *Journal of Applied Mechanics*, Vol. 26, No. 4, pp. 599–602, Dec., 1959.

During a symposium on plasticity at Brown University on April 5, 1960, under sponsorship of the Navy Research, a paper was presented by R. W. Haythornthwayte of the University of Michigan, titled "Stress and Strains in Soils," which presented experimental results indicating that the normality principle may not be valid. I would therefore suggest to use some caution in relying on these theories unless sufficient experimental work has proven them to be correct.

Although there are apparently still some problems to be solved with regard to the bulk solids flow and the measurement of the mechanical properties, the authors certainly deserve credit for having stimulated this new interest in bulk solids flow and the scientific approach.

MR. C. B. PUTNEY.[3]—We of Du pont now have a small research program which we call "Characterization of Bulk Solids." We are trying to define properties of bulk solids which would have meaning in the conveying or handling of the material.

Mr. Colijn suggested that bulk density can be used. Actually, however, there is no good definition for bulk density. For example, we produce a flake material which has a bulk density ranging from 6 lb per cu ft to 35 lb per cu ft depending on how much pressure has been used to pack it. When someone asks what the bulk density of this flake material is, we cannot tell them unless we give them some sort of a figure which has not been defined.

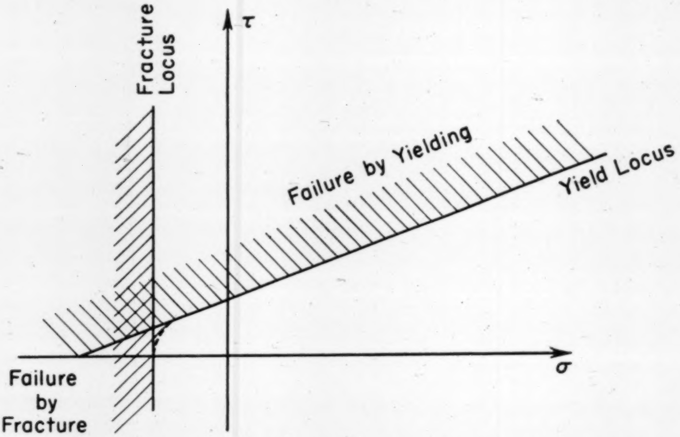


FIG. 22.—σ-τ Diagram for an Ideal Bulk Material.

The trade defines bulk solids as sticky, flaky, crumbly, dense, light—all of which are meaningless. We often receive calls from our plants: "I have a material to handle, and I need help." The first question in return is, "What is it like?" The answer is: "It is kind of like lime, sort of free flowing but kind of sticky too." So a sample is requested. Unfortunately, after receipt of the sample, little more is known about the material than before.

---

[3] Consultant, Engineering Service Division, Materials Handling, Packing, and Distribution Group, Engineering Department, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

Admittedly, after the sample is received, it can be examined to determine how it feels and appears, but actually very little engineering information can be obtained.

What I wish to point out is the fact that the research that Mr. Jenike has been doing has just broken the ice. We still need a tremendous amount of research in this field. Someone has to come up with some basic definitions which define the properties of bulk solids. We do not know how we are going to use these definitions, but the first thing we have to do is to formulate them. I picked up a short quotation from a magazine recently which I think states the problem. Lord Kelvin said in 1883, "When you can measure what you are speaking about and express it in numbers, you know something about it, but when you cannot measure it, you cannot express it in numbers, and your knowledge is of a meager and unsatisfactory kind. It may be the beginning of knowledge, but you have scarcely in your thoughts advanced to the state of science whatever the matter may be." That is a state we are now in. We are certainly not in a state of science for bulk solids handling.

I wish to say again that the author deserves all the support that we can all give him.

MR. W. S. HOUSEL[4] (*by letter*).—For two years the writer has been deeply interested in and somewhat concerned with the approach adopted in the authors' paper to describe the behavior of bulk solids. The interest stems from the fact that the flow of bulk solids is an important problem in many major industries; and, through their comprehensive experimental program, the authors have been making a substantial contribution to factual knowledge of the behavior of these materials. Secondly, their development of a theoretical approach is of direct interest to those in the field of soil mechanics, and it should be coordinated with related work by other investigators in the latter field. The writer's concern with this second phase of their work arises from the indication that theoretical development appears to be getting involved in the same confusion of basic concepts in the behavior of granular materials and granular-cohesive mixtures which has impeded progress in soil mechanics and led to a virtual stalemate in the development of certain fundamental aspects of soil resistance.

This concern is directed particularly to the basic properties of these materials and the concepts of behavior as formulated in definitions presented by the authors. When the paper was presented, the writer discussed this phase of the subject briefly from the floor to emphasize the vital importance of definitions, with the intent of later presenting a more carefully prepared written discussion. This has proved to be peculiarly perplexing, as such things frequently are, as it has been difficult for the writer to crystallize his thoughts and document the specific source of his misgivings.

Seeking help from Webster as to the meanings of "define" and "definition," one finds that to define is to "mark the limits of, to make definite and clear." A definition is an "explanation of the meaning . . . which fixes the limit of or restricts" the application of the definition, or that which serves to define, precisely, or bring into sharp relief. Definitions presented by the authors as a basis for their theoretical development and test procedure may be examined following Webster, thus providing a starting point for this discussion.

In the opening paragraph, the important definition is given by assuming that "a bulk solid is sufficiently closely approximated by a modified rigid-plastic Coulomb solid." To the writer, this is a definition which does the opposite of define; it might be called a claim for editorial license, permitting the authors to ascribe to bulk solids the classical properties of rigid solids, plastic solids, and so-called Coulomb materials, and then modify them at will. If this were not enough to remove all limits on the interpretation of test results, the opening paragraph also includes the statement: "Bulk solids exhibit elastic-plastic and viscous properties as well as a time effect similar to the jelling of Bingham materials and to the upper and lower yield points in mild steel."

---

[4] Professor of Civil Engineering, University of Michigan, Research Consultant, Michigan State Highway Department, Ann Arbor, Mich.

In the first place, the writer would question the wisdom of the practice, that appears to have developed from classical mechanics, of describing basic properties of materials by reference to individuals whose contributions in a certain field have been outstanding. Appropriate acknowledgment of their work is important, but is an independent matter and not part of precise thinking about fundamental principles. For example, if one examines Coulomb's original essay, it is found that basically he is dealing with a granular material which can be precisely defined in terms of the relationship of principal pressures which is clearly recognized in Coulomb's "wedge theory" of earth pressure and in all other classical earth pressure theories which follow. This statement does not rule out the recognition by Coulomb and others of the introduction of cohesion in granular-cohesive mixtures. In this connection, it is most important to differentiate clearly between these basically different properties in order to later deal with them precisely in their combined action. It is also interesting to note the meticulous care with which Coulomb prescribes the limits of the assumptions which he applied to the material with which he was dealing, limitations which seem to be completely forgotten in the later enunciation of the so-called Coulomb equation as a fundamental principle of mechanics.

In emphasizing the vital importance of definitions, it may be helpful to point again to the experience of investigators in the field of soil mechanics, to which previous reference has been made.[5] At the last two International Conferences on Soil Mechanics and Foundation Engineering, summation by General Reporters concluded that no agreement was possible, after some 35 years of research, on the most fundamental aspects of soil resistance. Casagrande implied that this came about from the failure to establish "more clearly defined basic concepts"; and, in calling for definitions, he said: "Most important among these basic elements will be a clear and simple definition of shear strength due to cohesion and shear strength due to internal friction."

Although there are some other elements involved, Casagrande's statement applies directly to the present discussion, which so clearly needs such definitions. In the hope of making a positive contribution to the discussion, the writer will present his understanding of the properties of bulk solids in the form of definitions for the record and for future discussion, if nothing more:

Granular materials are aggregates of solid particles which transmit pressure and develop resistance to displacement through intergranular forces and reactions acting between particles too large to be measurably affected by molecular forces.

The distinctive character of granular materials and their behavior under load result from the mechanical properties of the mass which may be summarized concisely in the definition of the two properties of internal friction and internal stability:

---

[5] W. S. Housel, "Dynamic and Static Resistance of Cohesive Soil—1846–1958," Soils—1959 Meetings, *ASTM STP No. 254*, Am. Soc. Testing Mats., p. 4 (1959).

> *Internal friction* is that mechanical property which provides resistance to tangential displacement through interlocking of grains, or particle interference, in which the shearing resistance is directly proportional to normal pressure.
>
> *Internal stability* is that mechanical property of granular materials which provides resistance to displacement under load through mutual support of adjacent particles in the mass.

The quantitative relation which identifies such granular materials is the proportionality maintained between principal pressures, expressed by a principal stress ratio. This ratio is constant for any specific granular material and mechanical arrangement of particles. It follows that changes in mechanical arrangement resulting in changes of the principal stress ratio produce, in effect, a different material, although it may be possible to deal with such changes as a continuous transition in the behavior of the original material. Such effects take place in most materials subjected to load, to a degree conditioned by the relative magnitude of the applied stress.

Changes of this nature are variously known as stress conditioning, strain hardening, and consolidation. The authors presumably classify materials which undergo such changes as "Bingham materials"; but it seems unnecessary to complicate the discussion of such well-known phenomena by a reference to some individual or his work, which may include other extraneous or poorly defined attributes. It would appear sufficient and less confusing to recognize these effects directly by the character and magnitude of the changes themselves.

If bulk solids investigated by the authors do exhibit the properties of granular-cohesive mixtures, it is essential to define cohesion with equal care and precision, delineating limits within which cohesion may apply:

> *Cohesion*, or shearing resistance due to cohesion, is that property of materials which provides finite static resistance to tangential displacement through molecular attraction between particles of a mass, characteristic of microscopic and submicroscopic matter. In contrast to internal friction, shearing resistance due to cohesion is independent of normal pressure, a relationship inherent in any material capable of sustaining a constant difference in principal pressures.

There are several points where the application of more precise definitions and a clearer concept of the behavior of granular materials may be helpful in dealing with bulk solids. The authors rely upon the geometry of the Mohr stress diagram to develop several concepts which turn out to be basic considerations in their development. In this connection, it should be pointed out that there is no implicit relation between propositions in geometry and the mechanics of force systems, although, obviously, geometry provides a useful and necessary means of formulating quantitative relations.

On the basis of measurements, the authors conclude that there are three modifications required in the Coulomb equation: first, a curved Mohr envelope or "yield locus"; second, a terminal point beyond which this locus does not extend; and, third, that the yield locus should go through the origin, representing that an unconsolidated, loose bulk solid has no strength at all.

The first modification, introducing a curved Mohr envelope, has been suggested by a number of investigators of granular materials to represent changes in the mechanical properties of the mass. While there are some unresolved questions to be answered, this modification is being generally accepted as consistent with experimental observations. Under stress conditioning by consolidation, the angle of internal friction is increased and the angle of failure is steeper. In terms of arching action, the angle of pressure transmission is steeper and the mechanical advantage supplied by particle arrangement also increases. It means, however, that we are no longer dealing with what the authors call a "Coulomb material"—one with a constant principal stress ratio. Under these conditions, the Coulomb equation is no longer valid, and its use in the geometry of the Mohr stress diagram may well lead to erroneous interpretation of test results.

The second modification, involving a fixed terminal point of the Mohr envelope, can be questioned. As a general property of granular materials, there seems to be no valid reason to presume that the relationship between principal pressures, expressed by the principal stress ratio, cannot be extended to any range of pressures which can be provided experimentally, up to the crushing strength of the grains. In the writer's opinion, such a terminal point is simply the reflection of a special test condition and represents the maximum reacting pressure which can be supplied by the weight of the granular mass or by external reactions provided.

The third modification, which, in effect, requires that the Mohr stress envelope or yield locus go through the origin, would be accepted by the writer as being entirely valid. Having established this point, however, there seems to be no logical reason for the lengthy speculation on the shape of the yield locus in the vicinity of the origin. To do so leads to several inconsistencies or basic errors which the writer finds most unacceptable.

The authors introduce failure in tension and use the term "hydrostatic tension" to describe this hypothetical stress in materials without cohesion and incapable of supplying tensile resistance. This results from failure to recognize that granular materials may expand in localized regions when the applied pressure exceeds the reacting pressure in the mass required for mutual support among particles. Such expansion is akin to stress-less volume change, frequently overlooked in dealing with internal equilibrium in relation to external reactions.

Another inconsistency into which the authors are led by their attempt to extend the Mohr envelope in terms of the Coulomb equation is an angle of internal friction, ϕ, equal to 90 deg. If this were true, the coefficient of friction or tangent of the angle of internal friction would be infinity. Such a material would not fail in shear and should support any applied pressure up to the crushing strength of the grains. This conclusion is similar to that which led the writer to reject internal friction and the Coulomb equation as a means of describing internal stability in granular materials.[6, 7] This led to the theory of arching action embodied in the definition of internal stability presented in this discussion.

To account for flow of bulk solids, the authors postulate a "free surface" on which the minor principal pressure is normal to the surface and equal to zero. Such a device voids the original assumption of proportionality between principal pressures which characterizes granular materials in the first place. The major principal pressure, f_c, is then called the "unconfined yield pressure," which must

---

[6] W. S. Housel, "Internal Stability of Granular Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. XXXVI, Part 2, p. 426 (1936).

[7] W. S. Housel, "Interpretation of Triaxial Compression Tests on Granular Mixtures," *Proceedings*, Assn. Asphalt Paving Technologists, Vol. XIX (1950).

correspond to the unconfined compressive strength introducing cohesion into the mixture. Later in the same paragraph it is said that f_c must be greater than zero to form a free surface, but it is still zero in a perfectly free-flowing solid for all degrees of consolidation. At this point, the writer must confess to being quite confused and unable to follow the authors' intent.

In conclusion, it would seem that there must be some way to describe the behavior of granular materials and bulk solids in logical and consistent terms which would reveal the true source of the mechanical properties of such masses. The writer would first suggest that the idea of arching action and mutual support be explored. In this way, the principal stress ratio may be expressed directly as a ratio of principal pressures, without reference to internal friction and the Coulomb equation and the complications which result. Finally, another attractive idea is presented by Weymouth's theory of particle interference which brings in gradation as a factor obstructing flow, which is consistent with the concept of internal stability as the product of mechanical arrangement and arching action.[8]

MESSRS. A. W. JENIKE, P. J. ELSEY, AND R. H. WOOLLEY (*authors' closure*).—By the time this material is printed in the *Proceedings*, a bulletin of the Utah Engineering Experiment Station, University of Utah, on "Gravity Flow of Solids" will have been published and some of the questions posed in this discussion will, it is the hope of the authors, have been answered at length.

In answer to Mr. Colijn's comments concerning the use of consolidating pressure rather than density as the independent variable in the measurement of the yield locus of a material, the authors' answer is as follows:

(*a*) Over the whole range of consolidating pressures, density changes very little and hence is insensitive as a variable.

(*b*) In the mathematical analysis which Jenike has developed (see: "Gravity Flow of Solids") and in the testing procedure it is more convenient to use consolidating pressure.

(*c*) Density does not uniquely define the yield locus of a material. Tests carried out at the Utah Engineering Experiment Station Bulk Solids Flow Laboratory as well as independently by others have shown that the method by which a given density has been attained significantly affects the yield locus of the solid. For instance, a solid can be consolidated by pounding, vibration, and hydrostatic pressure as well as under conditions of flow. But the last method most closely approaches the actual conditions in a bin.

With respect to Richmond's suggested yield loci, Figs. 21 and 22: they are essentially the same as those described in the paper, provided the filling in of the corners is given full status of a plane line and the sharp corners are erased. Sharp corners in a yield locus are meaningless and confusing to the unitiated.

So far as the principle of normality is concerned, the authors are unaware of any serious criticism of this principle. However, in the present work this principle is neither relied upon nor contradicted.

In reply to Mr. Putney, the authors humbly suggest that many of the answers he is seeking are already available in this paper and in Jenike's more recent publication.

Mr. Housel's comments concerning the definitions are quite pertinent. It is indeed a fact that in seeking a theoretical approach to the flow of bulk solids, the authors had to draw from the fields of rheology, plasticity, and soil mechanics. Each of these fields has established its own peculiar terminology. In addition, the authors have felt the need for additional concepts to which names had to be given. As a result, the subject has become specialized and difficult to follow. However, this problem is shared by all branches of technology and there is no easy solution to it.

---

[8] Weymouth, "Effect of Particle Interference in Mortars and Concrete," *Rock Products*, Feb. 25, 1933.

The authors prefer to use only such definitions as can be expressed quantitatively and that fit the fundamental equations of mechanics. No terms have been introduced that are not measurable and that are not directly applicable to the problems at hand.

Concerning the yield locus of a bulk solid, the authors have built on concepts already developed and have adapted them to suit their particular problem. As is plainly stated, the yield locus of a bulk solid is *not* identical with Coulomb's yield locus, and Mr. Housel's references to the Coulomb concept seem irrelevant.

Mr. Housel seems to deny the separate existence of the yield locus and of the effective yield locus. A careful reading of the text of the paper will bring out the important differences between those two concepts. The yield locus is the envelope of Mohr circles which represent stresses causing tensile (brittle) failure, or shear failure of a solid consolidated to a given density under conditions of flow. The effective yield locus is the envelope of Mohr circles which represent stresses causing flow of the solid for all degrees of consolidation.

Their geometric representations are different too. Thus, while the effective yield locus passes through the origin in the σ-τ coordinates, the yield loci (and, evidently, there is an infinite number of them for a given solid) does not necessarily do so. While the effective yield locus can well be approximated by a straight line, the yield loci, in most cases, cannot, and their shape has to be determined accurately because they define the flow-function of the solid.

To each degree of consolidation of a solid—as measured, for instance, by its density—there corresponds a yield locus with terminal points. No stress beyond the terminal points can be applied without consolidating the solid to a higher density and thus shifting to a new yield locus. Mr. Housel's conclusion that an angle of internal friction ϕ of 90 deg at the point of intersection of the yield locus with the σ-axis implies infinite shear strength is erroneous. At that point the shear strength is obviously zero and not infinity.

The authors are very grateful to the discussors for their interest in the paper and for their contributions.

A NOTCHED RING SPECIMEN FOR HYDROGEN EMBRITTLEMENT STUDIES*†

By F. S. Williams,[1] W. Beck,[2] and E. J. Jankowsky[3]

The restraining load on a stressed hydrogen-embrittled notched C-ring decreases with increasing crack growth, and the strain in a bolt used for stressing the specimen is reduced accordingly. Using this principle, a method of indicating crack propagation in a hydrogen-embrittled stressed ring specimen was developed. The advantages and disadvantages of the method are discussed, and its usability is demonstrated for the case of plating embrittlement.

It is often necessary to perform hydrogen embrittlement measurements under actual service conditions, for example, in the investigation of: the embrittlement of high-strength steel structural parts containing high residual stresses, during and after electroplating, during acid pickling, or during cathodic cleaning; the sulfide or sweet cracking of alloy steel tubing in oil wells or high-pressure petroleum pipe lines (**1**);[4] the possibility of brittle failures in cathodically protected underground steel structures (**2**); the embrittlement hazard in closed stainless steel containers exposed to high temperature, hydrogen, and pressure in chemical plants (**3**).

In some of these cases, periodic removal of test specimens is difficult if not impossible, and, consequently, laboratory methods such as determinations of bend and tensile ductility or delayed brittle failures are not suitable. For such applications, a notched C-ring was designed which is stressed by an instrumented bolt. The decrease in restraining load in the stressed embrittled rings as cracks propagate is reflected in a corresponding decrease in stress in the bolt. The decrease in bolt strain is sensed by a resistance strain gage attached to the bolt, and recorded.

The ring method can be used for measurements under service conditions where stress-rupture machines and other methods requiring bulky equipment are not applicable. Also, the method is remote-indicating, and strain changes can be recorded simultaneously in numerous specimens exposed at different locations.

The method is ideally suited for tubing and for testing sheet if the necessary forming can be permitted. Production of a ring specimen from bar stock, on the other hand, may be expensive. The inspection of the notch along its entire length may also cause some difficulty.

---

* Presented at the Sixty-third Annual Meeting of the Society, June 26–July 1, 1960.

† The opinions expressed are the private ones of the authors' and are not to be construed as official or reflecting the views of the Department of the Navy or the Naval Service at large.

[1] Superintendent, Metallurgical Division, Naval Air Material Center, Philadelphia, Pa.

[2] Head, Chemical Metallurgy Branch, Metallurgical Division, Naval Air Material Center, Philadelphia, Pa.

[3] Materials Engineer, Metallurgy Division, Naval Air Material Center, Philadelphia, Pa.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper.

The notched C-ring was described by the authors some time ago (4). Recently it has been used with success in the study of sulfide cracking of steels (1). The complete measuring system is described in this paper.

The utility of the method for determining plating embrittlement is demonstrated by comparing the notched ring results with those obtained on plated notched tension specimens subjected to sustained loading.

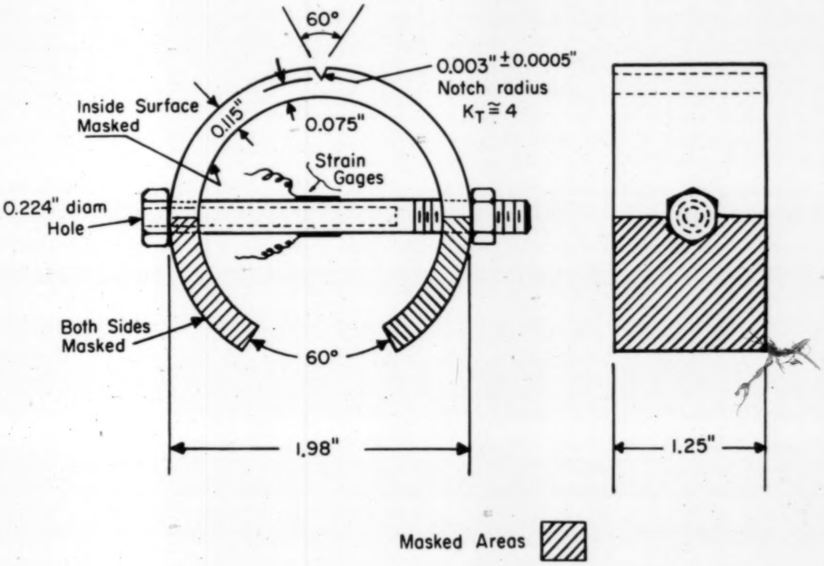

60°
0.003" ± 0.005"
Notch radius
$K_T \cong 4$
Inside Surface Masked
0.115"
0.075"
Strain Gages
0.224" diam Hole
Both Sides Masked
60°
1.98"
1.25"
Masked Areas ▨

FIG. 1.—Notched C-Ring With Instrumented Loading Bolt.

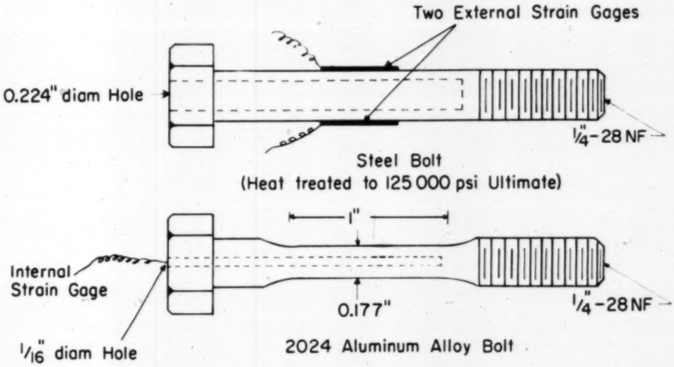

Two External Strain Gages
0.224" diam Hole
1/4"-28 NF
Steel Bolt
(Heat treated to 125 000 psi Ultimate)
1"
Internal Strain Gage
0.177"
1/4"-28 NF
1/16" diam Hole
2024 Aluminum Alloy Bolt

FIG. 2.—Types of Loading Bolts.

EXPERIMENTAL PROCEDURES

Calibration of Loading Bolts:

The notched C-ring specimens, henceforth called "rings," consisted of $1\frac{1}{4}$-in. sections cut from 2.0 in. diameter, $\frac{1}{8}$ in. wall tubing of AISI 4340 steel, heat treated to 260,000 to 280,000 psi ulti-

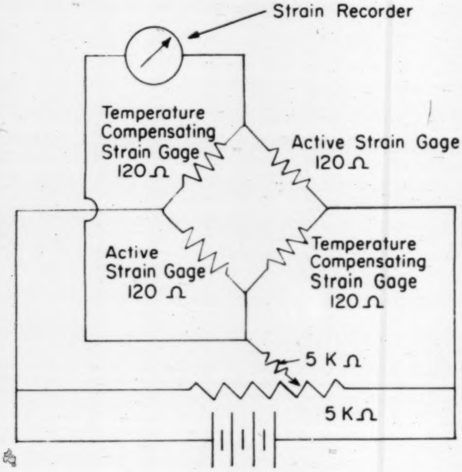

Strain Recorder
Temperature Compensating Strain Gage 120 Ω
Active Strain Gage 120 Ω
Active Strain Gage 120 Ω
Temperature Compensating Strain Gage 120 Ω
5 K Ω
5 K Ω

FIG. 3.—Strain-Gage Wiring Diagram.

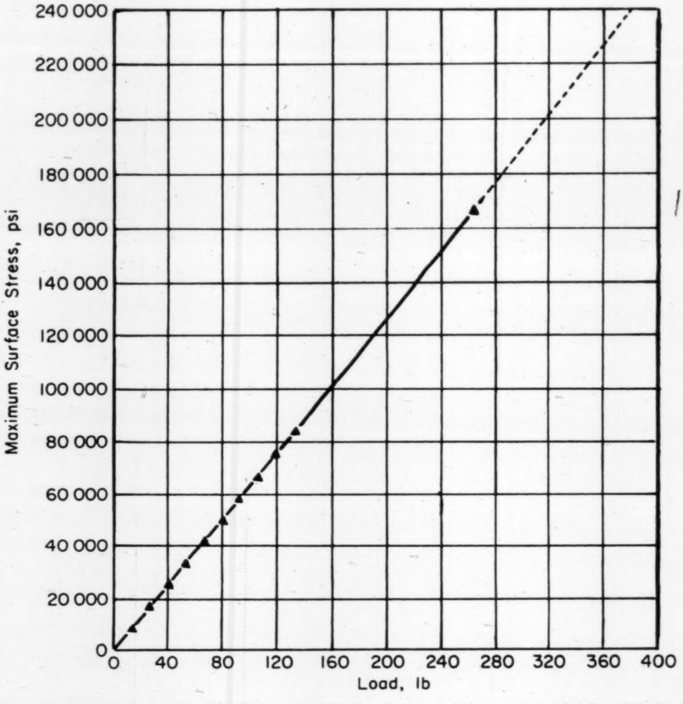


FIG. 4.—Ring Stress versus Applied Load, Unnotched C-Ring of 0.075 in. Wall Thickness.

mate (4). Notches were cut by careful grinding after heat treatment. Specimen dimensions are given in Fig. 1.

The rings were stressed by tightening a nut on the loading bolt. In the majority of the experiments, a $\frac{1}{4}$-28 AISI 2330 steel bolt was used, with two SR-4 type AD-7 strain gages attached. To increase the strain, the cross-sectional area of the bolt was reduced by drilling a 0.224-in. hole in its center as shown in Fig. 2.[5]

A limited number of measurements were made with a bolt of improved design (Fig. 2) which consisted of a $\frac{1}{4}$-28 bolt of 2024 aluminum alloy with its diameter reduced to 0.177 in. over a 1-in. length. A single strain gage was inserted in a $\frac{1}{16}$-in. hole drilled $1\frac{1}{2}$ in. deep in the center of the bolt. Identical results were obtained with both types of bolts. The advantages of the internally instrumented bolt are: (1) the strain gage is protected against damage; (2) the bolt can be readily reused; and (3) errors from bending are compensated for as effectively as with two gages on opposite sides.

---

[5] To prevent bending, point loading washers should be placed under the heads of the bolts.

The strain gages attached to the steel bolt formed two arms of a bridge circuit. The other arms consisted of two strain gages attached to a similar, unstrained bolt for temperature compensation. A schematic diagram of the electrical wiring is shown in Fig. 3.

The unbalance in the bridge circuit resulting from strain in the bolt was recorded by a galvonometer-type oscillograph, whose deflections were calibrated in terms of bolt stress. The chart paper was driven at 1 in. per min. Each of the bolts was calibrated in terms of strain produced by an applied load, and the strain was converted into bolt stress.

Calculation of Ring Stresses:

An unnotched C-ring of 0.075 in. wall thickness with a strain gage having a $\frac{1}{8}$-in. active length mounted at the center of its outside (tension) surface was loaded in compression in a universal testing machine, and the strains produced for various loads were recorded. The stresses corresponding to each of these strains were then calculated. Figure 4 is a plot of these values.

The load required to produce a given nominal stress in a notched ring is the same as that required to produce a surface stress of the same magnitude in an unnotched ring, provided the section moduli are the same. For rings of equal diameter, this means that the thickness of the unnotched ring must be the same as the thickness beneath the root of the notch in the notched ring. It is then

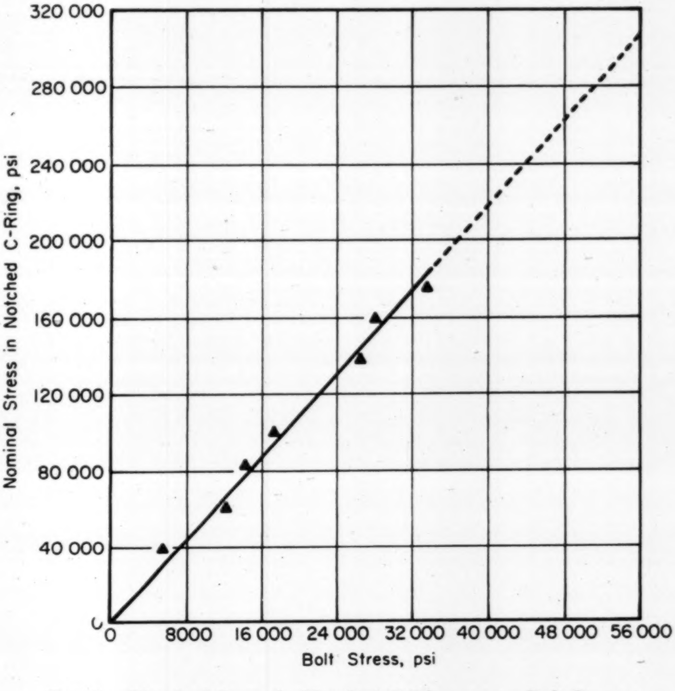


FIG. 5.—Nominal Stress in Notched C-Ring versus Bolt Stress.

possible to relate directly the maximum surface stresses obtained from loads on an unnotched ring, shown in Fig. 4, to nominal notched-ring stresses resulting from restraining bolt loads, and thus to bolt stresses.

To permit the convenient calculation of nominal stresses for specimens differing only by the depth of the notch and the thickness of the ring, a "corrected" value for the moment arm, l, was obtained by substituting the stresses calculated from the measured strains and the corresponding applied load, P, required to produce these stresses in the formula,

$$S = \frac{6Pl'}{bd^2} \quad \text{or} \quad l' = \frac{Sbd^2}{6P} \qquad (1)$$

where:

S = the maximum nominal stress parallel to the longitudinal axis of the beam at the root of the notch, calculated from measured strains,

P = the applied load,

l' = the moment arm "corrected" for specimen curvature,

d = the thickness of the cross-section at the root of the notch, and

b = the section width.

The nominal stresses in the ring produced by various loads, computed from Eq 1, were then plotted versus the corresponding bolt stresses. The linear nature of this plot is illustrated in Fig. 5. Bolt stresses determined from the oscillogram were converted to nominal stress in the ring by use of Fig. 5.

Stress Concentration and Static Notched Tensile Strength:

Depth and radius of the V-notch were selected to produce a high degree of stress triaxiality. The theoretical stress concentration factor ($K_T \cong 4$)[6] was obtained from Neuber's "Theory of Notch Stress" (**5**), for the case of a plate of finite thickness with a shallow notch subjected to bending. Specimens were rejected when the shadowgraphically measured notch dimensions were outside the permissible tolerances (±0.0005 in. for radius and ±0.001 in. for depth).

Due to machining difficulties, notches with radii below 0.003 in. could not be consistently ground into the rings.

The notch radius of the tension specimens used for the sustained-loading, delayed-failure tests was 0.002 in. (±0.0005 in.), and the theoretical stress concentration factor was found to be 6. The static notched tensile strength of both types of unplated specimens was approximately 315,000 psi.

Plating:

The notched C-ring was used to measure crack propagation induced by hydrogen in stressed electroplated steel. Three plating baths, cyanide cadmium, fluoborate cadmium, and chromium, were used for this investigation, because their embrittling characteristics were fairly well established, and results from this method could be compared with results from delayed-failure notched tension tests.

All specimens were anodically cleaned in a hot alkaline solution and plated with approximately 0.0005 in. of cadmium from the fluoborate or cyanide bath, or approximately 0.002 in. of chromium from the standard chromic-sulfuric acid bath. The plating thickness was measured on the unnotched portion of the specimen; the plating was restricted to the area under tensile stress by masking all other portions of the ring. Plating thickness at the base of the notch was about 50 per cent less than on the gage (**6,7**). All specimens were stressed 5 min after completion of plating.

---

[6] Based on the dimensions of the unplated notch.

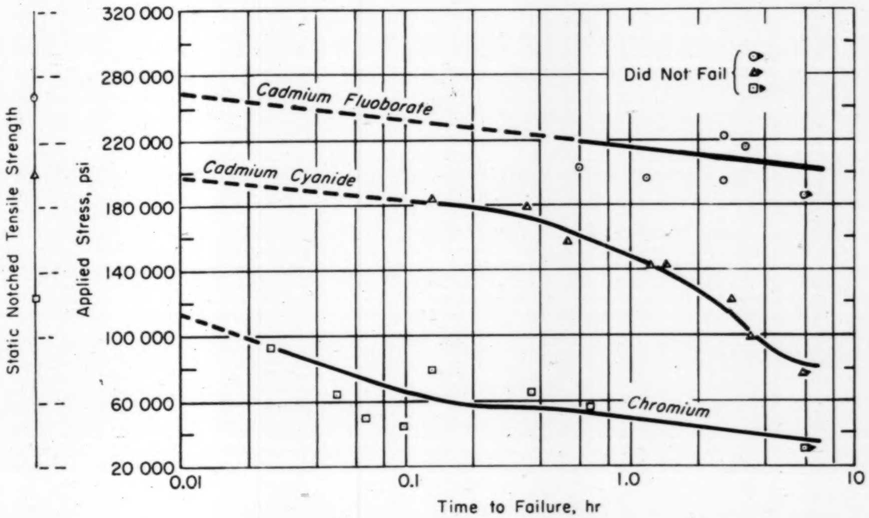


FIG. 6.—Delayed-Failure Curves for Plated Notched C-Rings.

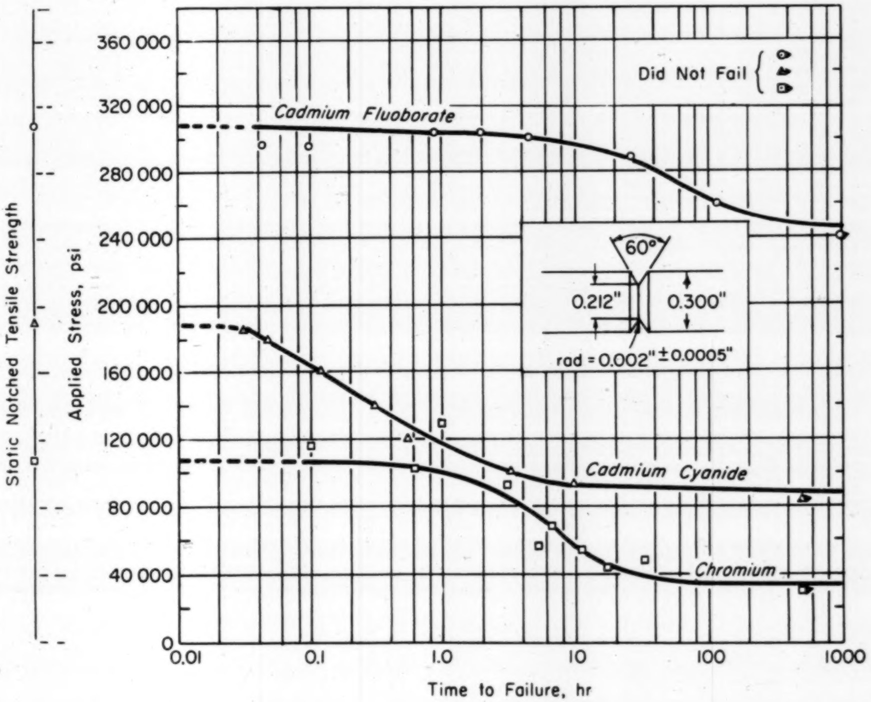


FIG. 7.—Delayed-Failure Curves for Plated Notched Tension Specimens.

TEST RESULTS

Figure 6 is a plot of the applied nominal ring stress versus time to failure. The graph shows well-defined sustained-loading delayed-failure curves with striking differences in the embrittlement behavior of the rings plated with cadmium from two different baths and with chromium.

A comparison of this plot with that of applied stress versus time to failure of the notched tensile bars (Fig. 7) is instructive. Both graphs demonstrate that the crack propagation rates in the plated stressed specimens increase in the order cadmium fluoborate, cadmium cyanide, and chromium. From these results, it may be concluded that the sensitivities of these two different types of specimens in indicating the effects of plating on hydrogen embrittlement and crack propagation rate, respectively, are quite similar.

The decrease in restraining-bolt stresses due to crack progression (Fig. 8) is also instructive. On a cadmium fluoborate plated ring, the time that elapsed before crack propagation started, as indicated by a rapid fall in the bolt stress, was comparatively long. This time interval, also called "incubation period" (8), indicates that the quantity of hydrogen introduced into the steel surface during plating is low.

In contrast, the almost instant decrease in bolt stress with the chromium-plated ring indicates a comparatively high surface concentration of hydrogen. With a steep concentration gradient, hydrogen diffuses rapidly into the steel, a process that results in a rapid rate of crack propagation and a comparatively short time to failure (9).

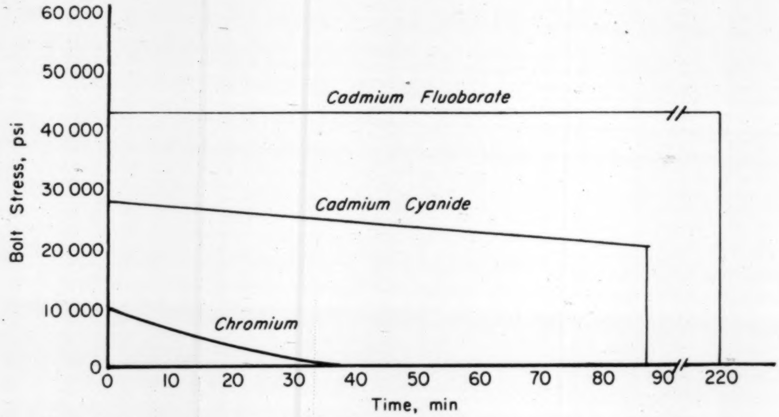


FIG. 8.—Crack Progression in Plated Notched C-Ring as Indicated by Decrease in Loading Bolt Stress.

CONCLUSIONS

The notched C-ring used with an instrumented loading bolt and a strain recorder represents a useful, sensitive method for measuring hydrogen embrittlement. This method appears to be adaptable to other applications requiring a small, comparatively simple, remote-indicating device for the assessment of various types of cracking under service conditions.

Acknowledgment:

The authors are much indebted to Mr. Samuel Goldberg, Special Material Section of the Bureau of Naval Weapons, for the interest taken in this study and for numerous stimulating discussions.

REFERENCES

(1) R. L. McGlasson and W. D. Greathouse, "Stress Corrosion Cracking of Oil Country Tubular Goods," *Corrosion*, Vol. 15, p. 437t (1959).

(2) W. H. Bruckner and K. M. Myles, "Studies on the Susceptibility of Cathodically Protected Steel to Hydrogen Embrittlement," *Corrosion*, Vol. 15, p. 591t (1959).

(3) D. D. Perlmutter and B. F. Dodge, "Effect of Hydrogen on Properties of Metals," *Industrial Engineering Chemistry*, Vol. 48, p. 885 (1956).

(4) W. Beck and E. J. Jankowsky, "Effect of Plating High Tensile Strength Steels," *Proceedings*, Am. Electroplaters' Soc., Vol. 45, p. 57 (1958).

(5) H. Neuber, "Theory of Notch Stresses," J. W. Edwards Co., Ann Arbor, Mich., p. 181 (1946).

(6) E. Raub, "Research on Microthrowing Power and Leveling of Plating Baths," *Plating*, Vol. 45, p. 489 (1958).

(7) E. P. Klier, B. B. Muvdi, and George Sachs, "The Response of High Strength Steels in the Range of 180,000 to 300,000 psi to Hydrogen Embrittlement from Cadmium-Electroplating," *Proceedings*, Am. Soc. Testing Mats., Vol. 58, p. 598 (1958).

(8) A. R. Troiano, "Delayed Failure of High Strength Steels," *Corrosion*, Vol. 15, p. 207t (1959).

(9) H. H. Johnson, J. G. Morlet, and A. R. Troiano, "Hydrogen, Crack Initiation, and Delayed Failure in Steel," *Transactions*, Metallurgical Soc., Am. Inst. Mining and Metallurgical Engrs., Vol. 212, p. 528 (1958).

DISCUSSION

MR. J. W. KAUFMAN[1] (*presented in written form*).—This paper is valuable in that it provides a standardized, fully instrumented testing technique for the measurement of the effects of hydrogen embrittlement by mechanical means. Modifications of the ring technique have been utilized by many investigators. A ring and other test methods have been described by Vlannes, Strauss, and Brown, in their paper, "The Problem of Fracture of Ultra-High-Strength Steel Electroplated with Cadmium."[2] Raring and Rinebolt describe a test method using a notch tension specimen and a dead weight lever arm type of machine in their article "Static Fatigue of High Strength Steel."[3] However, the test described in the paper discussed here appears to be capable of closer control and greater duplication and does not require the tying up of elaborate testing equipment or the need to have such equipment at all.

In addition, the instrumentation appears most reliable and the test results therefrom highly reproducible. This test, therefore, should be considered with a view to making it an industry standard.

MR. ARTHUR B. WILDER.[4]—Considerable work has been conducted, as the authors have stated, on C-ring testing of oil-well tubing with outside diameters of $2\frac{3}{8}$ in. and $2\frac{7}{8}$ in. This work has been essentially related to hydrogen embrittlement and stress in wells with a corrosive environment. In addition to hydrogen embrittlement, the subject of stress-corrosion cracking of the branching type has also been investigated. Comments of the authors on the following items will be appreciated.

---

[1] Metallurgical Engineer, Radio Corp. of America, Camden, N. J.

[2] *Proceedings*, Symposium on Materials Research in the Navy, U. S. Naval Research Laboratory, *ONR 5*, Vol. 2 (1959).

[3] *Transactions*, Am. Soc. Metals, Vol. 48, p. 198 (1956).

[4] Chief Metallurgist, National Tube Division, U. S. Steel Corp., Pittsburgh, Pa.

1. Are the C-ring test results with a notch in the longitudinal direction similar to the notched tension test results with a notch in the transverse direction?

2. Are the delayed failure curves for zinc and tin coatings similar to those for cadmium coatings?

3. Are the lower-strength materials less susceptible to hydrogen embrittlement as compared with the high-strength materials?

4. Have any studies in the C-ring test been made of unnotched, hot-rolled surfaces and compared with unnotched-machined surfaces?

MR. WALTER BECK (*author*).—With respect to the comparison between test results from C-rings with a longitudinal notch and tension specimens with a transverse notch, it may be stated that the trends of sustained loading curves are the same for both types of specimens and both approach the same "static fatigue limit" for identical embrittling treatments. However, failure takes place very much faster in notched C-rings than in notched tension specimens at equivalent stresses.

Inasmuch as the theoretical stress concentration factor is lower for the ring than for the tension specimen, the more rapid failure of the notched ring is somewhat unexpected. However, Darken[5] has demonstrated that the driving force for diffusion is an activity gradient for the diffusing element. In this case, it may be speculated that the activity gradient for the ring is greater than for the notched tension specimen due to the greater stress gradient in the ring resulting from the bending stresses.

In answer to the second question, sustained loading delayed failure tests of zinc- or tin-plated specimens have not been performed at the Naval Air Material Center. It is believed, however, that the curves for these coatings would be similar to those for cyanide cadmium coatings since it has been reported that AISI 440-C stainless steel wire (ultimate tensile strength 133,000 psi) is strongly embrittled when plated with zinc from the cyanide bath. Embrittlement from the acid zinc bath was shown to appreciably lower.[6] AISI 4340 steel (ultimate tensile strength 260,000 to 280,000 psi) is also strongly embrittled when it is cyanide zinc-plated. Plated notched tension specimens subjected to sustained loading at 50 per cent of the unplated notched tensile strength do not show any response to baking, even when the baking time is extended to 200 hr at 350 F.[7] AISI 440-C and 4340 steel when plated with tin from the alkaline stannate bath are also strongly embrittled.[8,9]

As to the third question, based on MIL Spec. QQ-P-416a (Dec. 11, 1956), "Plating Cadmium, Electrodeposited," and studies of the bend ductility of cyanide cadmium-plated AISI 4340 steel conducted at the Naval Air Material Center, the statement may be made that hydrogen embrittlement is not considered significant below a Rockwell hardness, C scale, of 40 (approximately 180,000 psi ultimate tensile strength).

Finally, no studies of unnotched, hot rolled C-ring surfaces compared with machined surfaces have been conducted at the Naval Air Material Center.

---

[5] L. S. Darken, "Diffusion of Carbon in Austenite with a Discontinuity in Composition," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 180, p. 430 (1949).

[6] C. A. Zappfe and M. E. Haslem, "Hydrogen Embrittlement in Cadmium and Zinc Electroplating," *Plating*, Vol. 37, p. 366 (1950).

[7] B. F. Brown, "Effect of Baking on Delayed Fracture of Electroplated Ultra High Strength Steel," *Naval Research Laboratory Report No. 4839*, Oct. 3, 1956.

[8] C. A. Zappfe and M. E. Haslem, "Note on Embrittlement from Alkaline Tin Plating," *Plating*, Vol. 39, p. 468 (1952).

[9] W. Beck and E. J. Jankowsky, "Effect of Plating High Tensile Strength Steel," *Proceedings*, Am. Electroplaters' Soc., Vol. 44, p. 49 (1957).

MR. BERTRAM FLEISCHER.[10]—In the graphs showing stress versus time-to-failure the tests are carried out to only 10 hr for the C-ring type specimen whereas they are carried to 1000 hr for the notched tension type specimen.

Could the authors give an idea as to how long a test should be run to determine whether or not hydrogen embrittlement is present, and what stress level should be used, particularly for material whose ultimate strength is about 260,000 psi?

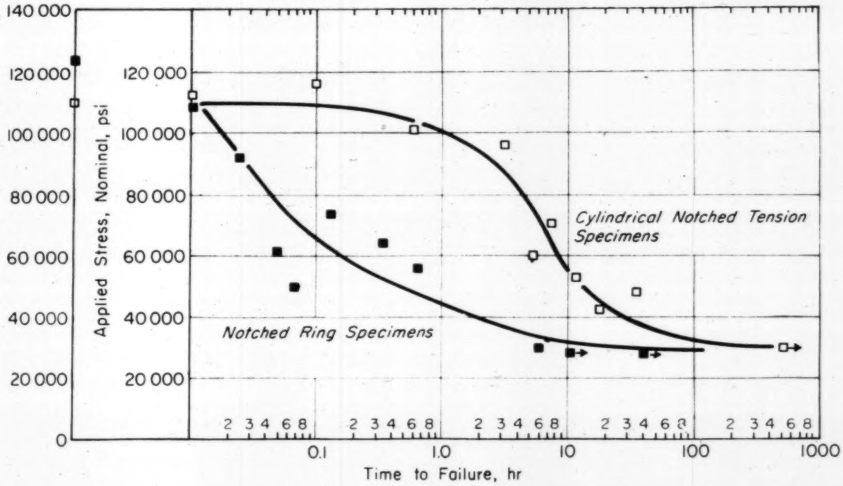


FIG. 9.—Comparison of the Behavior of Two Types of Specimens Plated with Chromium to a Thickness of 0.002 in.

MR. F. S. WILLIAMS (*author*).—The choice of stress level and time for detecting hydrogen embrittlement is somewhat arbitrary. Various organizations use different criteria, the length of time a test should be run being determined by the stress level applied.

We have normally started out at about 90 per cent of the yield strength and then worked downward to determine these curves. Your observation concerning the difference in time on the two types of tests is a good one. We normally would continue testing until the line becomes horizontal—that is, until it appears we have reached the stress level which would give infinite life. We are doing additional work presently on the notched ring to carry the tests out to longer times, as shown in the accompanying Fig. 9. The graph also illustrates the striking differences in failure times of different types of specimens stressed to the same level.

MR. R. F. DECKER.[11]—Do you have any feeling on the effectiveness of these coatings or other coatings on hydrogen pick-up in service—not the hydrogen in electroplating but from the environment in service?

MR. WILLIAMS.—I would expect cadmium to be effective as a barrier and the cadmium-tin alloy type of coating to be somewhat superior to cadmium alone. I would expect that certain types of chromium platings would be relatively poor as barrier coatings, particularly in the case of the chromium coatings which are cracked.

We have conducted experiments in which we attempted to diffuse hydrogen through the coating and have found that cadmium was quite effective in preventing entry of hydrogen. We have tried flashes of cadmium and then a subsequent bake, to produce a barrier, prior to putting on the regular thickness or coating. This approach has been partially effective.

---

[10] Design Metallurgist, Convair, Fort Worth, Tex.

[11] Senior Metallurgist, The International Nickel Co., Inc., Bayonne, N. J.

MR. R. KOPITUK.[12]—Does this confirm the work that Troiano[13] published some time back on the effectiveness of the double plating with the hydrogen embrittlement relief in between?

MR. WILLIAMS.—Yes, there is certainly something to it. There is, however, in this whole matter of the ultra-high-strength steels no real assurance that we are completely free of hydrogen embrittlement. We reduce the susceptibility of delayed failures under sustained loadings, but we are never certain that we eliminate them entirely at high stress levels.

---

[12] Manager, Metallurgy and Materials, Thiokol Chemical Corp., Inc., Reaction Motors Division, Denville, N. J.

[13] H. H. Johnson, E. J. Schneider, and A. Troiano, "New Technique Rids Plated Steel of Hydrogen Embrittlement," *Iron Age*, July 21, 1958.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON RADIATION EFFECTS AND RADIATION DOSIMETRY

This Symposium on Radiation Effects and Radiation Dosimetry presents some of the latest research on the effects of radiation on materials properties as well as discussions of parameters to be considered in predicting and evaluating such effects. Also presented are newer techniques for radiation dosimetry.

The symposium, sponsored by ASTM Committee E-10 on Radioisotopes and Radiation Effects, was held during the Eighteenth and Twenty-second Sessions of the Sixty-third Annual Meeting of the Society, on Wednesday, June 29, 1960.

Mr. C. G. Collins, General Electric Co., acted as Symposium Chairman and presided over the morning session. Vice-President M. N. Clair, the Thompson and Lichtner Co., Inc., presided over the afternoon session.

The paper by A. D. Rossin was not presented during the symposium but was added to the published volume because of its interest.

The papers included in the symposium are:

Introduction—C. G. Collins
Determinant Parameters in Radiation Effects Testing of High Polymers—E. G. Fritz
Irradiation Testing of Enrico Fermi Prototype Fuel Pins in the CP-5 Reactor—M. A. Silliman, A. A. Shoudy, P. R. Huebotter, and W. G. Blessing
Effect of Neutron Irradiation on Charpy V and Drop-Weight Test Transition Temperatures of Various Steels and Weld Metals—J. R. Hawthorne and L. E. Steele
Reactor Spectra Considerations in Radiation Effects Predictions—J. Romanko
Radiochemistry and Mass Spectrometry in Fuel Depletion Studies—J. E. Hudgens, Jr.
Effect of Sample Parameters on Energy Absorption from a Neutron Beam—R. L. Johnston
High-Intensity Gamma Ray Dosimetry—D. Hale, D. R. Johnson, S. M. Dec, J. R. Coss, O. V. P. Sessoms, P. B. Hemmig, W. L. R. Rice, and R. E. Brocklehurst
A Solid State Bragg-Gray Cavity Chamber—Victor H. Ritz and Frank H. Attix
Measurement of the Energy Absorbed from Pile Neutrons—D. Binder, C. D. Bopp, and R. L. Towns
Neutron Dosimetry for Materials Irradiation Studies—L. E. Steele and J. R. Hawthorne
Neutron Spectrum Determinations at Low Flux Levels—Paul Kruger
Radiation Damage in Steel: Considerations Involving the Effect of Neutron Spectra—A. D. Rossin

These papers with discussion were issued as *ASTM Special Technical Publication No. 286* entitled "Symposium on Radiation Effects and Radiation Dosimetry."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON QUALITY OF OBSERVATIONS

The purpose of this Symposium is to discuss some fundamental problems in the interpretation of measurements. The quality of a result is often given by statements about the precision and accuracy. The evaluation of the precision and accuracy requires a collection of test results obtained under conditions encountered in practice. The four papers in this Symposium attempt to clarify the meanings of these terms and to survey methods that can be used to determine precision and accuracy.

The papers and discussions in this Symposium were presented at the Thirty-fifth Session of the Sixty-third Annual Meeting of the Society, held on June 30, 1960, in Atlantic City, N. J. The Symposium was sponsored jointly by the Administrative Committee on Research and Committee E-11 on Quality Control of Materials.

Mr. A. T. McPherson, Associate Director, National Bureau of Standards acted as symposium chairman and presided over the Session.

The papers included in the Symposium are:

On the Meaning of Precision and Accuracy—R. B. Murphy
How to Evaluate Precision—W. S. Connor
How to Evaluate Accuracy—W. J. Youden
On the Analysis of Planned Experiments—Milton E. Terry

The papers were published in *Materials Research & Standards*, April, 1961, Vol. 1, No. 4, pp. 264–277.

SUMMARY OF PROCEEDINGS OF CHICAGO, ILL. SPRING MEETING

During the 1960 Committee Week held in Chicago, Ill., a panel discussion was held on February 3, constituting the 1960 Spring Meeting:

PANEL DISCUSSION ON APPEARANCE OF ALUMINUM SURFACES

This panel discussion was sponsored by Subcommittee 4 of Committee E-12 on Appearance. Arrangements for the meeting were made by R. V. Paulson, Kaiser Aluminum and Chemical Corp. who also served as moderator.

The main purpose in arranging this panel discussion was to discuss problems associated with the matching of high-silicon architectural aluminum products when they are anodized to shades of gray. Difficulty in matching the appearance of such anodized products has been experienced by major aluminum producers, fabricators, and architects. Each producer has a matching procedure for use or guidance of his customers. These procedures are similar but trouble arises because they are not identical. It was accordingly felt that since there were no standard procedures for measuring or specifying appearance of metal surfaces with colored transparent or semi-transparent coatings, an effort should be made to establish some. Such methods would be found very useful in the automotive and appliance industries and in other industries where a control of color and appearance is a production problem.

In addition to Mr. Paulson, the other panel members and the subjects which they discussed were as follows:

Mr. R. Myer—Metallurgical Manager, Kaiser Aluminum and Chemical Corp., Oakland, Calif., discussed the importance of appearance measurement from the management viewpoint.

Mr. Eric F. Barkman—Research Supervisor, Reynolds Metals Co., Richmond, Va., discussed gloss measurements of aluminum surfaces.

Mr. W. C. Cochran—Assistant Chief, Finishes Division, Aluminum Company of America Research Laboratories, New Kensington, Pa., discussed the color measurement of transparent and semi-transparent colored films on aluminum.

Brief summaries of this panel discussion appear in the ASTM BULLETIN No. 242, December, 1959, p. 33, and No. 245, April 1960, p. 46.

1960 TECHNICAL PAPERS PUBLISHED IN ASTM BULLETIN

ASTM Bulletin *No. 243, January 1960*

A Study of the Accelerated Aging of Vinyl Plastic Compounds in a Modified Testing Oven—Markus Royen, p. 43 (TP 1)

A New Calibration Technique for Gas Transmission in Measuring Apparatus—T. McAvoy, p. 52 (TP 10)

Control Testing for Separation of Lightweight Material from Aggregate—E. C. Higginson and G. B. Wallace, p. 60 (TP 18)

A Practice Method for Classifying All Elastomeric Vulcanizates—N. L. Catton, R. C. Edwards, and T. M. Loring, p. 69 (TP 27)

The Langley as a Unit for Timing Outdoor Exposures—R. Caryl, p. 55 (TP 13)

ASTM Bulletin *No. 244, February 1960*

Machining of Notched Tension Test Specimens—J. L. March, W. J. Ruprecht, and George Reed, p. 52 (TP 56)

An Instrument for Determination of Impact Sensitivity of Materials in Contact with Liquid Oxygen—William R. Lucas and Wilbur A. Riehl, p. 29 (TP 33)

High Temperature Vacuum Equipment for Treatment of Reactive and Refractory Metals—A. L. Feild, Jr., and V. A. Peckham, Jr., p. 34 (TP 38)

Hardness, Abrasion, and Wear Resistance Testing of Plastics—Ladislav Boor, p. 43 (TP 47)

A Reliable Technique for Measuring Brush Wear in an Atmosphere Containing Silicone Vapor—J. S. Axelson and F. M. Precopio, p. 40 (TP 44)

ASTM Bulletin *No. 245, April 1960*

Tests for Potential and Past Moisture Expansion of Ceramic Building Units—E. H. Waters, J. S. Hosking, and H. V. Huber, p. 55 (TP 61)

Measurement for Environmental Stress-Cracking of Polyethylene—A. Rudin and A. M. Birks, p. 60 (TP 66)

An Alignment Block and Clamp for Capillary Viscometers—W. M. Robinson, p. 69 (TP 75)

The Use of 2,2,4,4,6,8,8-Heptamethylnonane as a Primary Diesel Fuel—T. W. Mears, R. M. David, and F. L. Howard, p. 75 (TP 81)

Discussion of Paper on Control Testing for Separation of Lightweight Material from Aggregate—W. G. Hime, p. 78 (TP 84)

Movement of Sodium with Water in Neat Portland Cement—C. L. Ford, p. 71 (TP 77)

ASTM Bulletin *No. 246, May 1960*

*An Improved Microhardness Tester for High Temperature Use—J. H. Westbrook, p. 53 (TP 113)

Measurement of Surface Moisture and Sulfur Dioxide Activity at Corrosion Sites—P. J. Sereda, p. 47 (TP 107)

A Technique for Gripping High-Strength Fabrics During Physical Tests—Karl F. Plett and Lee A. Dunlap, p. 33 (TP 93)

---

* Presented at the Sixty-third Annual Meeting June 26–July 1, 1960.

Design Criteria for 6Al-4V Titanium Alloy at Room and Elevated Temperatures—M. M. Lemcoe and J. K. Childs, p. 41 (TP 101)

* An Improved Method for the Determination of the Normal Consistency of Gypsum Plasters—R. A. Kuntze, p. 35 (TP 95)

A Vicat-Type Penetration Tester for Evaluating Hardness and Elastic Recovery in Polymeric Materials—K. W. Gardiner, T. F. Jordan, and F. W. Adams, p. 38 (TP 98)

Rheological Testing of Elastomers at Low Temperatures—L. Boor, M. Conant, and W. Scoville, Jr., p. 25 (TP 85)

Measurement of the Brittleness Temperature of Polyethylene—A. M. Birks and A. Rudin, p. 49 (TP 109)

* An Improved Microhardness Tester for High-Temperature Use—J. H. Westbrook, p. 53 (TP 113)

ASTM Bulletin *No. 247, July 1960*

The Tensile Properties of Some Engineering Materials at Moderate Rates of Strain—Robert F. Steidel, Jr., and Cyril E. Makerov, p. 57 (TP 123)

Measurement of Human Reaction to Hardness of Floor Coverings—R. Schjödt, p. 53 (TP 119)

Effect of Dispersion Technique Upon the Measured Particle Size and Distribution of Tungsten Powder—A. I. Michaels, T. L. Weaver and R. C. Nelson, p. 74 (TP 141)

Creep Characteristics of Compression-Molded Polyethylene—G. R. Gohn and J. D. Cummings, p. 64 (TP 131)

A Gravimetric Method for the Determination of Barium Oxide in Cement—C. L. Ford, p. 77 (TP 143)

ASTM Bulletin *No. 248, September 1960*

Devices for Simulating Gun Firing Under Static Conditions—J. P. Noonan, R. W. Heinemann, S. J. Lowell, and P. B. Tweed, p. 33 (TP 147)

The Measurement of Relative Density of Sand—Shigeru Kitago and Fumiwo Kozake, p. 36 (TP 154)

Development of Improved Insulating Oils—N. W. Furby and F. J. Hanly, p. 41 (TP 155)

A Test Method for Air-Entrainment of Standard Ottawa Sand—Martin R. DeFore and Harold J. Corah, p. 48 (TP 162)

Measuring the Roughness Factor of a Small Steel Disk—C. Begovich, p. 56 (TP 166)

Additional Variables in the Brittleness Temperature Testing of Polyethylene—G. L. Bata and M. Ito, p. 55 (TP 165)

ASTM Bulletin *No. 249, October 1960*

Determination of the Thermal Stability of Disiloxane-Base Hydraulic Fluids—Myra Willard, p. 52 (TP 196)

A High-Temperature, Vacuum, Axial Fatigue Testing Machine—R. I. Stephens and G. M. Sinclair, p. 44 (TP 188)

Lightfastness Testing for Pigment Colors—W. F. Spengemen and G. Wormald, p. 29 (TP 173)

Water Permeability Studies on Latex and Latex Paint Films—Roy C. Simon, p. 48 (TP 192)

Moisture Penetration of Brick Masonry Panels—T. Ritchie and W. G. Plewes, p. 39 (TP 183)

Low-Temperature Tensile-Hardness Correlations for SAE 4340 Steel—J. Nunes and F. R. Larson, p. 25 (TP 169)

Flexural and Impact Variations of Phenolic Moldings: A Statistical Round-Robin Study—C. Elmer and E. C. Harrington, Jr., p. 35 (TP 179)

ASTM Bulletin *No. 250, December 1960*

Punch Card Handling of Atmospheric Test Data—W. H. Ailor and M. R. Hodgson, p. 45 (TP 223)

A More Sensitive Test Method for Determining the Dielectric Strength of Insulating Oils—P. F. Ast, p. 36 (TP 214)

Properties of Some Copper Alloys at Elevated Temperatures—C. H. Crowe, p. 30 (TP 208)

Use of a Modified MacMichael Viscometer for Consistency of Printing Inks and Ink Vehicles—R. Cummings, p. 49 (TP 233)

The Permeability of Sand Stabilized with a Chemical Grout—G. H. Dorion, H. Burkhard, and M. White, p. 34 (TP 212)

A Short Method for the Flame Photometric Determination of Magnesium, Manganic, Sodium, and Potassium Oxides in Portland Cement—C. L. Ford, p. 25 (TP 203)

* A New Impact Detonability Tester for Evaluating Material with Highly Reactive Oxidizers—Kopituk, p. 51 (TP 229)

————1960 ANNUAL MEETING PAPERS PUBLISHED IN 1961————

Materials Research & Standards, *Vol. 1, No. 1, January 1961*

Formerly ASTM BULLETIN

* The Evaluation of Resistance Strain Gages at Elevated Temperatures—R. L. Bloss, p. 9

To Be Published

* Shock Testing of Ground Support Equipment and Industrial Items Weighing from 1,000 to 50,000 Pounds—W. H. Grumet

*SYMPOSIUM ON QUALITY OF OBSERVATIONS

On the Meaning of Precision and Accuracy—R. B. Murphy, Vol. 1, No. 4, April
How to Evaluate Precision—W. S. Connor, Vol. 1, No. 4, April
Some Observations on Evaluating Accuracy—W. J. Youden, Vol. 1, No. 4, April
On the Analysis of Planned Experiments—Milton Terry, Vol. 1, No. 4, April

AUTHOR INDEX

1960 TECHNICAL PAPERS AND COMMITTEE REPORTS

A

Adam, L. H. and Dougherty, M.

Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings. Report of Committee B-7 (Appendix II), 186.

Adams, F. W.

See Gardiner, K. W., Jordan, T. F., and Adams, F. W.

Ahlvin, Richard G. and Brown, Donald N.

Duplication of Prototype Stress-Strain Relations in Soil Masses by Laboratory Tests, 1137. Disc., 1149.

Ailor, W. H., and Hodgson, M. R.

Punch Card Handling of Atmospheric Test Data, Published in ASTM BULLETIN, No. 250, Dec., 45 (TP 223).

Anderson, Elaine R.

See Kantro, D. L., Copeland, L. E. and Anderson, Elaine R.

Anderson, R. L.

Automatic Polishing of Metallographic Specimens—Summary of Proceedings of *STP 285*, 1003.

Anderson, W. J.

See Zaretsky, E. V., and Anderson, W. J.

Ashby, G. E.

Index to the Literature on X-Ray Spectrographic Analysis. Part I, 1913–1957. *STP 292*.

Ast, P. F.

A More Sensitive Test Method for Determining the Dielectric Strength of Insulating Oils, Published in ASTM BULLETIN, No. 250, Dec., 36 (TP 214).

Attix, F. H.

See Ritz, V. H. and Attix, F. H.

Aubrey, Eric

Discussion, 601.

Axelson, J. S., and Precopio, F. M.

A Reliable Technique for Measuring Brush Wear in an Atmosphere Containing Silicone Vapor, Published in ASTM BULLETIN, No. 244, Feb., 40 (TP 44).

B

Bader, W. G.

See Gohn, G. R., and Bader, W. G.

Baker, Robert F.

Discussion, 1149.

Baker, W. O.

The Meaning of Measurement, ACR notes, Published in ASTM BULLETIN, No. 245, April, 42.

Bata, G. L., and Ito, M.

Additional Variables in the Brittleness Temperature Testing of Polyethylene, Published in ASTM BULLETIN, No. 248, Sept., 55 (TP 165) (Technical Note).

Batchelder, G. M.

Discussion, 666.

Bazett, D. J. and Smotrych, S. W.

The Compressive Strength of Remolded Niagara Clay, 1124. Disc., 1132.

Beck, W.

See Williams, F. S., Beck, W., and Janowsky, E. J.

Begovich, C.

Measuring the Roughness Factor of a Small Steel Disk, Published in ASTM BULLETIN, No. 248, Sept., 56 (TP 166) (Technical Note).

Belton, J. H., Godby, L. L., and Taft, B. L.

Materials for Use at Liquid Hydrogen Temperatures—Summary of Proceedings of *STP 287*, 1004.

Benjamin, I. A. and Ratliff, G. D., Jr.

The Effects of Early Freezing of Low-Density Aggregate-Type Concrete, 1080.

Bennett, J. A.

See Frankel, H. E., Bennett, J. A., and Carman, C. M.

Bernett, E. C.

Discussion, 956.

Bickford, W. B.

See Raville, M. E., Bickford, W. B., and Huber, D. A.

Binder, D., Bopp, C. D. and Towns, R. L.

Measurement of the Energy Absorbed from Pile Neutrons—Summary of Proceedings of *STP 286*, 1203.

Birks, A. M.

See Rudin, A. and Birks, A. M.

Birks, A. M., and Rudin, A.

Measurement of the Brittleness Temperature of Polyethylene, Published in ASTM BULLETIN. No. 246, May, 49 (TP 109).

Blessing, W. G.

See Silliman, M. A., Shoudy, A. A., Huebotter, P. R., and Blessing, W. G.

Bloem, D.

Discussion, Published in ASTM BULLETIN, No. 243, Jan., 68 (TP 26).

Bloss, R. L.

Evaluation of Resistance Strain Gages at Elevated Temperaures, 1207.

Boor, L.

Hardness, Abrasion, and Wear Resistance Testing of Plastics, Published in ASTM BULLETIN, No. 244, Feb., 43 (TP 47). (*R*).

Boor, L., Hanok, M., Conant, F., and Scoville, W., Jr.

Rheological Testing of Elastomers at Low Temperatures. Part II, Published in ASTM BULLETIN, No. 246, May, 25 (TP 85). (*R*).

Bopp, C. D.

See Binder, D., Bopp, C. D., and Towns, R. L.

Borneman, P. R.

Discussion, 784.

Bramer, S. E.

Discussion, 538, 984.

Brandenburg, C. G.

Present Methods of Metallographic Specimen Preparation for Retention and Identification of Inclusions in Steel—Summary of Proceedings of *STP 285*, 1003.

Brock, G. W. and Sinclair, G. M.

Elevated-Temperature Tensile and Fatigue Behavior of Unalloyed Arc-Cast Molybdenum, 530. Disc., 538.

Brocklehurst, R. E.

See Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

Brooks, Harvey

Accomplishments and Limitations of Solid State Theory—Summary of Proceedings of *STP 283*, 499.

Brothers, A. J.

Discussion, 733.

Brown, Donald N.

See Ahlvin, Richard G. and Brown, Donald N.

Brown, W. F., Jr.

Discussion, 717.
See Epsey, G. B., Jones, M. H., and Brown, W. F., Jr.
See Shannon, J. L., Jr., Epsey, G. B., Repko, A. J., and Brown, W. F., Jr.

Brunberg, W. C.

Standardization and the Purchaser, Published in ASTM BULLETIN, No. 249, Oct., 6.

Bryan, E. L.

Photoelastic Evaluation of the Panel Shear Test for Plywood—Summary of Proceedings of *STP 289*, 1002.

Burcham, Jay F.

Preliminary Evaluation of Thermocouple Attachment Methods for Radiant Heating Applications, 957, Disc., 971.

Burkhard, H.

See Dorion, G. H., Burkhard, H., and White, M.

Burn, K. N.

Design and Calibration of a Neutron Moisture Meter—Summary of Proceedings of *STP 293*, 1166.

Burns, R.

In Defense of Committees, Published in ASTM BULLETIN, No. 246, May, 24.

C

Cain, F. M., Jr.

Simplified Metallographic Techniques for Nuclear Reactor Materials—Summary of Proceedings of *STP 285*, 1003.

Campbell, J. E. and Rice, L. P.

Properties of Some Precipitation-Hardening Stainless Steels and Low-Alloy High-Strength Steels at Very Low Temperatures—Summary of Proceedings of *STP 287*, 1004.

Carey, W. N., Jr., Shook, J. F., and Reynolds, J. F.

Evaluation of Nuclear Moisture-Density Testing Equipment—Summary of Proceedings of *STP 293*, 1166.

Carlton, P. F.

Application of Nuclear Soil Meters to Compaction Control for Airfield Pavement Construction—Summary of Proceedings of *STP 293*, 1166.

Carman, C. M.

Discussion, 783.
See Frankel, H. E., Bennett, J. A. and Carman, C. M.

Carpenter, J. A.

Stress Deformation Characteristics of Sand-Bituminous Mixtures—Summary of Proceedings of *STP 294*, 1167.

Caryl, C. R.

The Langley as a Unit for Timing Outdoor Exposures, Published in ASTM BULLETIN, No. 243, Jan., 55 (TP 13). (*R*).

Cataldo, C. E.

See Lucas, W. R. and Cataldo, C. E.

Catton, N. L., Edwards, R. C., and Loring, T. M.

A Practical Method of Classifying All Elastomeric Vulcanizates, Published in ASTM BULLETIN, No. 243, Jan., 69 (TP 27).

Childs, J. K.

See Lemcoe, M. M. and Childs, J. K.

Christian, J. L.

See Watson, J. F. and Christian, J. L.

Clapper, R. B.

Discussion, 955.
Shear and Torsion Testing of Solid Materials—A Critical Discussion—Summary of Proceedings of *STP 289*, 1002.

Clarke, F. E.

Old Wine in New Bottles. A New Look at the Substance, Water, Published in ASTM BULLETIN, No. 243, Jan., 17.

Clauss, Francis J.

An Examination of High-Temperature Stress-Rupture Correlating Parameters, 905. (*R*).

Coffin, L. F., Jr.

Discussion, 509.

Coffman, Bonner

Introduction, Symposium on Nuclear Soil Density and Moisture—Summary of Proceedings of *STP 293*, 1166.

Colijn, H.

Discussion, 1182.

Collins, C. G.

Introduction, Symposium on Radiation Effects and Radiation Dosimetry—Summary of Proceedings of *STP 286*, 1203.

Conant, F.

See Boor, L., Hanok, M., Conant, F., and Scoville, W., Jr.

Conner, W. S.

How to Evaluate Precision, Summary of Proceedings of the Symposium on Quality of Observation. Published in *Materials Research & Standards* (formerly ASTM BULLETIN), Vol. 1, No. 4, April, 1961, 263. (*See also*, 1204.)

Copeland, L. E.

See Kantro, D. L., Copeland, L. E., and Anderson, Elaine R.

Copson, H. R.

Long-time Atmospheric Corrosion Tests on Low Alloy Steels, 650 (*R*). Disc., 666.

Corah, H. J.

See DeFore, M. R., and Corah, H. J.

Coss, J. R.

See Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

Coyne, L. D.

See Mertens, E. W., Coyne, L. D., and Rogers, E. D.

Crooks, R. D.

Discussion, 881.
Discussion, Published in ASTM BULLETIN, No. 243, Jan., 54 (TP 12).

Cross, H. C.

Discussion, 866.

Crowe, C. H.

Properties of Some Copper Alloys at Elevated Temperatures, Published in ASTM BULLETIN, No. 250, Dec., 30 (TP 208).

Cummings, H. N.

Discussion, 510.

Cummings, J. D.

See Gohn, G. R., and Cummings, J. D.

Cummings, R.

Use of a Modified MacMichael Viscometer for Consistency of Printing Inks and Ink Vehicles, Published in ASTM BULLETIN, No. 250, Dec., 49 (TP 227).

Cummins, E. W.

Discussion, Published in ASTM BULLETIN, No. 243, Jan., 68 (TP 26).

D

Dalzell, R. C.

Nuclear Fuel Element Development (Gillett Lecture). (*Issued as a separate pamphlet.*)

Danek, G. J., Jr.

Discussion, 559.

Daniels, Farrington

Utilization of Solar Energy (Marburg Lecture). (*Issued as a separate pamphlet.*)

David, C. K.

Discussion, 788.

David, R. M.

See Mears, T. W., David, R. M., and Howard, F. L.

Dean, W. J.

Discussion, 973.

Dec, S. M.

See Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

Decker, R. F.

Discussion, 1201.

DeFore, M. R., and Corah, H. J.

A Test Method for Air-Entrainment of Standard Ottawa Sand, Published in ASTM BULLETIN, No. 248, Sept., 48 (TP 162).

DeMoney, F. W.

Discussion, 538, 971.

Dohr, A. W.

See Wood, Lyman, W., Erickson, E. C. O., and Dohr, A. W.

Dorion, G. H., Burkhard, H., and White, M.

The Permeability of Sand Stabilized with a Chemical Grout, Published in ASTM BULLETIN, No. 250, Dec., 34 (TP 212).

Dougherty, M.

See Adam, L. H. and Dougherty, M.

Douglas, D. A.

See Kennedy, C. R., and Douglas, D. A.

Drykacz, W. W.

Discussion, 780.

Dunlap, L. A.

See Plitt, K. F., and Dunlap, L. A.

E

Edwards, R. C.

See Catton, N. L., Edwards, R. C., and Loring, T. M.

Ellis, W. H. and Schmidt, R. J.

A Method for Measuring Air-Permeabilities of Asphalt Concrete Pavements—Summary of Proceedings of *STP 294*, 1167.

Elmer, C. and Harrington, E. C., Jr.

Flexural and Impact Variations of Phenolic Moldings: A Statistical Round-Robin Study. Published in ASTM BULLETIN, No. 249, Oct., 35 (TP 179).

Elsey, P. J.

See Jenike, A. W., Elsey, P. J., and Woolley, R. H.

Epsey, G. B.

See Shannon, J. L. Jr., Epsey, G. B., Repko, A. J., and Brown, W. F. Jr.

Epsey, G. B., Jones, M. H., and Brown, W. F. Jr.

Sharp-Edge-Notch Tensile Characteristics of Several High-Strength Titanium Sheet Alloys at Room and Cryogenic Temperatures —Summary of Proceedings of *STP 287*, 1004.

Erickson, E. C. O.

See Wood, Lyman W., Erickson, E. C. O., and Dohr, A. W.

F

Feild, A. L., Jr., and Peckham, V. A., Jr.

High-Temperature Vacuum Equipment for Treatment of Reactive and Refractory Metals, Published in ASTM BULLETIN, No. 244, Feb., 34 (TP 38).

Feltner, C. E.

See Sinclair, G. M. and Feltner, C. E.

Fenn, R. W., Jr.

Compression Testing of Sheet Magnesium Utilizing Rapid Heating, 940 (*R*). Disc., 955.

Discussion, 972.
Introduction, Symposium on Shear and Torsion Testing—Summary of Proceedings of *STP 289*, 1002.
Low Temperature Properties of Cast and Wrought Magnesium Alloys—Summary of Proceedings of *STP 287*, 1004.

Fischler, J. E.
See Tiner, N. A., and Fischer, J. E.

Fleischer, Bertram
Discussion, 1200.

Focht, J. A., Jr.
Discussion, 1120.

Ford, C. L.
A Gravimetric Method for the Determination of Barium Oxide in Portland Cement, Published in ASTM BULLETIN, No. 247, July, 77 (TP 143).
A Short Method for the Flame Photometric Determination of Magnesium, Manganic, Sodium, and Potassium Oxides in Portland Cement, Published in ASTM BULLETIN, No. 250, Dec., 25 (TP 203).
The Gravimetric Determination of Strontium Oxide in Portland Cement, Published in ASTM BULLETIN, No. 245, April, 71 (TP 77).

Forney, D. M., Jr.
Experimental Techniques and Equipment for Acoustical Fatigue Research and Development—Summary of Proceedings of *STP 284*, 1001.

Fox, A.
See Gohn, G. R., and Fox, A.

Frankel, H. E., Bennett, J. A. and Carman, C. M.
Fatigue Properties of some High Strength Steels, 501. Disc., 509.

Freeman, James
See Voorhees, Howard B. and Freeman, James W.

Freudenthal, A. M.
Fatigue of Structural Metals Under Random Loading—Summary of Proceedings of *STP 284*, 1001.
The Phenomenon of Stress-Relaxation, 986. (*R*). Disc., 1000.

Freytag, N. A.
Load Transfer in Spot Welds: A Preliminary Investigation with Photoelastic Coating—Summary of Proceedings of *STP 289*, 1002.

Fritz, E. G.
Determinant Sample Parameters in Radiation Effects Testing of High Polymers—Summary of Proceedings of *STP 286*, 1203.

Furby, N. W. and Hanly, F. J.
Development of Improved Insulating Oils, Published in ASTM BULLETIN, No. 248, Sept., 41 (TP 155) (*R*).

G

Gardiner, K. W., Jordan, T. F., and Adams, F. W.
A Vicat-Type Penetration Tester for Evaluating Hardness and Elastic Recovery in Polymeric Materials, Published in ASTM BULLETIN, No. 246, May, 38 (TP 98).

Garofalo, F.
Resistance to Creep Deformation and Fracture in Metals and Alloys—Summary of Proceedings of *STP 283*, 499.
Temperature Dependence on the Elastic Moduli of Several Stainless Steels, 738 (*R*).

Gerberich, W. W.
See Sachs, G., Gerberich, W. W., Weiss, V., and Latorre, J. V.

Gilman, John J.
Dislocation Motions and the Yield Strengths of Solids—Summary of Proceedings of *STP 283*, 499.

Gnaedinger, J. P.
Experiences with Nuclear Moisture and Density Surface Probes on O'Hare Field Project—Summary of Proceedings of *STP 293*, 1166.

Godby, J. G.
See Belton, J. H., Godby, J. B., and Taft, B. L.

Gohn, G. R. and Bader, W. G.
The Tensile Properties of Hard-Drawn Alloy A Phosphor Bronze Rod, 843.

Gohn, G. R., and Cummings, J. D.
Creep Characteristics of Compression-Molded Polyethylene, Published in ASTM BULLETIN, No. 247, July, 64 (TP 130).

Gohn, G. R. and Fox, A.
The R. R. Moore Rotating-Beam Fatigue Test, 560 (*R*). Disc., 568.

Goldhoff, R. M.
Discussion, 1000.

Gray, R. J.
See Long, E. J. Jr., and Gray, R. J.

Greeson, R. L.
See Reed, R. P., Mikesell, R. P., and Greeson. R. L.

Grover, H. J.
Discussion, 510, 538.

H

Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

High-Intensity Gamma Ray Dosimetry—Summary of Proceedings of *STP 286*, 1203.

Hall, R. C., and Rhodes, J. M.

Movement of Sodium with Water in Neat Portland Cement, Published in ASTM BULLETIN, No. 245, April, 66 (TP 72).

Hamaker, J. C., Jr.

Discussion, 778, 823.

Hamaker, J. C., Jr. and Vater, E. J.

Carbon: Strength Relationships in Five Per Cent Chromium Ultra-High-Strength Steels 691 (*R*). Disc., 717.

Hanly, F. J.

See Furby, N. W., and Hanly, F. J.

Hanok, M.

See Boor, L., Hanok, M., Conant, F., and Scoville, W., Jr.

Hanson, M. P., Stickley, W. G., and Richards, H. T.

Sharp-Notch Behavior of Some High-Strength Sheet Aluminum Alloys and Welded Joints at 75, −320 and −423 F—Summary of Proceedings of *STP 287*, 1004.

Harrington, E. C., Jr.

See Elmer, C. and Harrington, E. C., Jr.

Harris, J. C.

Metal Cleaning Bibliographical Abstracts—1960 Supplement—*STP 90-E*.

Methods for Analysis of Synthetic Detergents—Bibliographical Abstracts—1959 Supplement—*STP 150-C*.

Hawthorne, J. R.

See Steele, L. E. and Hawthorne, J. R.

Hawthorne, J. R. and Steele, L. E.

Effect of Neutron Irradiation on Charpy-V and Drop-Weight Test Transition Temperatures of Various Steels and Weld Metals—Summary of Proceedings of *STP 286*, 1203.

Heger, J. J.

Discussion, 687, 893.

Heinemann, R. W.

See Noonan, J. P., Heinemann, R. W., Lowell, S. J., and Tweed, P. B.

Hemmig, P. B.

See Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

Higginson, E. C., and Wallace, G. B.

Control Testing for Separation of Lightweight Material from Aggregate, Published in ASTM BULLETIN, No. 243, Jan., 60 (TP 18). Disc., 68 (TP 26).

Hime, W. G.

Discussion, Published in ASTM BULLETIN, No. 245, April, 78 (TP 84).

Hobstetter, John N.

Mechanical Properties of Semiconductors—Summary of Proceedings of *STP 283*, 499.

Hodgson, M. R.

See Ailor, W. H., and Hodgson, M. R.

Hosking, J. S.

See Waters, E. H., Hosking, J. S., and Hueber, H. V.

Housel, W. S.

Discussion, 1185.

Howard, F. L.

See Mears, T. W., David, R. M., and Howard, F. L.

Huber, D. A.

See Raville, M. E., Bickford, W. B., and Huber, D. A.

Hudgens, J. E., Jr.

Radiochemistry and Mass Spectrometry in Fuel Depletion Studies—Summary of Proceedings of *STP 286*, 1203.

Hueber, H. V.

See Waters, E. H., Hosking, J. S., and Hueber, H. V.

Huebotter, M. A.

See Silliman, M. A., Shoudy, A. A., Huebotter, P. R., and Blessing, W. G.

Hull, Frederick C.

Effects of Alloying Additions on Hot-Cracking of Austenitic Chromium-Nickel Stainless Steels, 667 (*R*). Disc., 686.

Hurlich, A.

Summation, Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials—Summary of Proceedings of *STP 287*, 1004.

Hveem, F. N.

ASTM Standards for Highway Materials, Published in ASTM BULLETIN, No. 249, Oct., 8.

I

Irwin, G. R.

Discussion, 736.
Effects of Size and Shape on Fracture of Solids—Summary of Proceedings of *STP 283*, 499.

Ito, M.

See Bata, G. L., and Ito, M.

J

Janowsky, E. J.

See Williams, F. S., Beck, W., a Jandnowsky E. J.

Jenike, A. W., Elsey, P. J. and Woolley, R. H.

Flow Properties of Bulk Solids, 1168. Disc., 1184.

Johnson, D. R.

See Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

Johnson, W. H. and Kura, J. A.

Mechanical and Physical Properties of Five Copper-Base Casting Alloys, 796. Disc., 811.

Johnson, W. R.

Discussion, 881.

Johnston, R. L.

Effect of Sample Parameters on Energy Absorption from a Neutron Beam—Summary of Proceedings of *STP 286*, 1203.

Jones, E. S.

Discussion, 788.

Jones, M. H.

Discussion, 717.
See Epsey, G. B., Jones, M. H., and Brown, W. F. Jr.

Jordan, T. F.

See Gardiner, K. W., Jordan, T. F., and Adams, F. W.

K

Kantro, D. L., Copeland, L. E., and Anderson, Elaine R.

An X-Ray Diffraction Investigation of Hydrated Portland Cement Pastes, 1020 (*R*). Disc., 1035.

Kaplan, M. F.

The Reproducibility of Flexure and Compression Tests for Determining the Strength of Cement, 1006 (*R*). Disc., 1018.

Kattus, J. R.

Discussion, 955, 971.

Kattus, J. R. and McDowell, D. W., Jr.

Strength of Structural Alloys Under Rapid Heating and Loading, 928.

Kaufman, J. W.

Discussion, 1199.

Kennedy, C. R. and Douglas, D. A.

Relaxation Characteristics of Inconel at Elevated Temperatures, 867 (*R*). Disc., 881.

Kennedy, T. B.

Discussion, 1062.

Kenney, T. C.

Discussions, 568, 1132.

Kiefer, R. W.

The Effect of Compaction Temperature on the Properties of Bituminous Concrete—Summary of Proceedings of *STP 294*, 1167.

Kies, J. A.

Discussion, 719, 789.

Kitago, S., and Kozaki, F.

The Measurement of Relative Density of Sand, Published in ASTM BULLETIN, No. 248, Sept., 36 (TP 150). Disc., 40 (TP 154).

Klier, E. P.

Discussion, 784.
Effect of Carbon Content on the Notch Properties of 43XX-Vanadium Modified, and Five Per Cent Chromium Sheet Steels—Summary of Proceedings of *STP 287*, 1004.

Kopituk, R.

A Method for Determining the Impact Sensitivity Characteristics of Materials with Highly Reactive Oxidizers, Published in ASTM BULLETIN, No. 250, Dec., 51, (TP 229) (Disc.).
Discussion, 1202.

Kozaki, F.

See Kitago, S., and Kozaki, F.

Kruger, Paul

Neutron Spectrum Determinations at Low Flux Levels—Summary of Proceedings of *STP 286*, 1203.

Kuenzi, E. W.

See Youngquist, W. G. and Kuenzi, E. W.

Kuntze, R. A.

An Improved Method for the Determination of the Normal Consistency of Gypsum Plasters, Published in ASTM BULLETIN, No. 246, May, 35 (TP 95).

Kura, J. A.

See Johnson, W. H., and Kura, J. A.

L

Landgren, Robert

See Verbeck, George, and Landgren, Robert.

Larson, F. R.

See Nunes, J. and Larson, F. R.

Latorre, J. V.

See Sachs, G., Gerberich, W. W., Weiss, V., Latorre, J. V.

Lazan, B. J.

Introduction, Symposium on Acoustical Fatigue—Summary of Proceedings of *STP 284*, 1001.
See Trapp, W. J. and Lazan, B. J.

Lee, H.

Compatibility of Magnet Wire Insulations and Epoxy Encapsulating Resins, Published in ASTM BULLETIN, No. 247, July, 69 (TP 135) (*R*).

Lehrer, William M. and Schwartzbart, Harry

Static and Fatigue Strength of Metals Subjected to Triaxial Stresses, 610 (*R*).

Lemcoe, M. M., and Childs, J. K.

Design Criteria for 6 Al-4V Titanium Alloy at Room and Elevated Temperatures, Published in ASTM BULLETIN, No. 246, May, 41 (TP 101) (*R*).

Lessells, John M.

Discussion, 509.

Levitt, Albert P. and Martin, Anthony G.

Application of Induction Heating to Short-Time Elevated-Temperature Tension Testing, 974. Disc., 984.

Link, H. S.

Mechanical Polishing of Metallographic Specimens—Summary of Proceedings of *STP 285*, 1003.

Long, E. J., Jr., Meador, J. T., and Gray, R. J.

Experience with Vibratory Polishers and Design for Cell Application—Summary of Proceedings of *STP 285*, 1003.

Loring, T. M.

See Catton, N. L., Edwards, R. C., and Loring, T. M.

Lowell, S. J.

See Noonan, J. P., Heinemann, R. W., Lowell, S. J., and Tweed, P. B

Lucas, W. R. and Cataldo, C. E.

Some Low-Temperature Properties of Aluminum Alloy Weldments—Summary of Proceedings of *STP 287*, 1004.

Lucas, W. R., and Riehl, W. A.

An Instrument for Determination of Impact Sensitivity of Materials in Contact with Liquid Oxygen, Published in ASTM BULLETIN, No. 244, Feb., 29 (TP 33).

Lunsford, L. R.

Bonded Metal-to-Metal Shear Testing—Summary of Proceedings of *STP 289*, 1002.

M

Makerov, C. E.

See Steidel, R. F., Jr., and Makerov, C. E.

Mann, J. Y.

The Fatigue-Notch Sensitivity of Annealed Copper, 602 (*R*). Disc., 607.

March, J. L., Ruprecht, W. J., and Reed, G.

Machining of Notched Tension Test Specimens, Published in ASTM BULLETIN, No. 244, Feb., 52 (TP 56).

Mark, H. F.

New Polymers—New Problems (Thirty-third Edgar Marburg Lecture), Published in ASTM BULLETIN, No. 245, April, 31.

Martin, Anthony G.

See Levitt, Albert P., and Martin, Anthony G.

Mather, B.

Discussion, Published in ASTM BULLETIN, No. 243, Jan., 68 (TP 26).
Discussion, 1061.

Mather, K.

Discussion, 1035.

Mayne, C. R.

Effect of Zerolling on Properties of Modified Type 347 Stainless Steel—Summary of Proceedings of *STP 287*, 1004.

McAvoy, T.

A New Calibration Technique for Gas Transmission Measuring Apparatus, Published in ASTM BULLETIN, No. 243, Jan,. 52 (TP 10).

McCaffrey, T. F.

Discussion, 784.

McConnaughay, K. E.

Emulsified Asphalt Tests and Specifications—Summary of Proceedings of *STP 294*, 1167.

McCoy, P.

Sound Standards Stimulate Manufacturing, Published in ASTM BULLETIN, No. 248, Sept. 5.

McDowell, D. W., Jr.

See Kattus, J. R., and McDowell, D. W., Jr.

McPherson, A. T.

Plan for the Self-Qualification of Laboratories, Published in ASTM BULLETIN, No. 246, May, 17.

What Price Accurate Test Methods? (ACR notes), Published in ASTM BULLETIN, No. 244, Feb. 7.

Meador, J. T.

See Long, E. J., Jr., Meador, J. T. and Gray, R. J.

Mears, T. W., David, R. M., and Howard, F. L.

The Use of 2,2,4,4,6,8,8-Heptamethylnonane as a Primary Diesel Fuel, Published by ASTM BULLETIN, No. 245, April, 75 (TP 81) (*R*).

Mertens, E. W.

Predicting Weatherability of Coating Grade Asphalts from Asphaltene Characteristics, Published in ASTM BULLETIN, No. 250, Dec., 40 (TP 218).

Mertens, E. W., Coyne, L. D., and Rogers, E. D.

Cationic Asphalt Emulsions—Recommended Tests and Specifications—Summary of Proceedings of *STP 294*, 1167.

Metzger, M. C.

See Simmons, Ward and Metzger, M. C.

Michaels, A. I., Weaver, T. L., and Nelson, R. C.

Effect of Dispersion Techniques Upon the Measured Particle Size and Distribution of Tungsten Powder, Published in ASTM BULLETIN, No. 247, July, 74 (TP 140).

Mikesell, R. P.

See Reed, R. P., Mikesell, R. P., and Greeson, R. L.

Miller, O. O.

Discussion, 893, 971.

Mintzer, Sidney

Comparison of Nuclear and Sand-Cone Methods of Density and Moisture Determinations for Four New York State Soils—Summary of Proceedings of *STP 293*, 1166.

Morrow, JoDean

Discussion, 644.

Moyer, G. J.

Discussion, 644.

Murphy, R. B.

On the Meaning of Precision and Accuracy, Summary of Proceedings of the Symposium on Quality of Observation. Published in *Materials Research & Standards* (formerly ASTM BULLETIN), Vol. 1, No. 4, April 1961, 263. (*See also*, 1204.)

Muvdi, B. B.

Structural Beryllium, 812 (*R*). Disc., 823.

N

Narrow, I.

Performance Tests of Field Concrete Mixers, 1047. Disc., 1061.

Neifert, Harry R.

Discussion, 643.

Nelson, R. C.

See Michaels, A. I., Weaver, T. L., and Nelson, R. C.

Nelson, R. C., and Rhodes, R. H.

Testing Machine Attachment Permits Straightness Measurements on Fine Wire, Published in ASTM BULLETIN, No. 244, Feb., 56 (TP 60) (Technical Note).

Neville, O. K.

Design and Application of the Nuclear Chicago d/M Gage—Summary of Proceedings of *STP 293*, 1166.

Noonan, J. P., Heinemann, R. W., Lowell, S. J., and Tweed, P. B.

Devices for Simulating Gun Firing Under Static Conditions, Published in ASTM BULLETIN, No. 248, Sept., 33 (TP 147).

Norton, Mary R.

Introduction, Symposium of Methods of Metallographic Specimen Preparation—Summary of Proceedings of *STP 285*, 1003.

Nunes, J. and Larson, R.

Low-Temperature Tensile-Hardness Correlations for SAE 4340 Steel, Published in ASTM BULLETIN, No. 249, Oct., 25 (TP 169).

O

O'Kelly, B. M.

Portable Adhesion Testing Device, Published in ASTM BULLETIN, No. 250, Dec., 32 (TP 210).

Olden, E. C.

Use of Diamond Abrasives in Metallographic Problems—Summary of Proceedings of *STP 285*, 1003.

P

Pall, Francis

Discussion, 781.

Parker, Earl R.

Status of Ductile Ceramic Research—Summary of Proceedings of *STP 283*, 499.
The Role of Refractory Metals in Superalloys, 849 (*R*). Disc., 866.

Peckham, V. A., Jr.

See Feild, A. L., Jr., and Peckham, V. A., Jr.

Penner, E.

The Importance of Freezing Rate in Frost Action in Soils, 1151 (*R*).

Peterson, R. E.

Discussion, 511.

Plewes, W. G.

See Ritchie, T. and Plewes, W. G.

Plitt, K. F., and Dunlap, L. A.

A Technique for Gripping High-Strength Fabrics During Physical Tests, Published in ASTM BULLETIN, No. 246, May, 33 (TP 93).

Precopio, F. M.

See Axelson, J. S., and Precopio, F. M.

Putney, C. B.

Discussion, 1185.

R

Radd, F. J.

Discussion, 666.

Randall, P. N.

Creep-Rupture Properties of a Carbon Steel and a Low Alloy Steel at 1200 to 1800 F, 885. Disc., 893.

Ratliff, G. D., Jr.

See Benjamin, I. A., and Ratliff, G. D., Jr.

Raville, M. E., Bickford, W. B. and Huber, D. A.

A Dynamic Test for Determining Shear Modulus of Sandwich Core Materials—Summary of Proceedings of *STP 289*, 1002.

Reed, G.

See March, J. L., Ruprecht, W. J., and Reed, G.

Reed, R. P., Mikesell, R. P., and Greeson, R. L.

Some Mechanical Properties of Magnesium Alloys at Low Temperatures—Summary of Proceedings of *STP 287*, 1004.

Reinhart, Fred M.

Twenty-Year Atmospheric Corrosion of Zinc-Coated and Uncoated Wire and Wire Products. (*Issued as a separate pamphlet STP 290.*)

Repko, A. J.

See Shannon, J. L., Jr., Epsey, G. B., Repko, A. J., and Brown, W. F., Jr.

Reynolds, G. M.

Discussion, 1118.

Reynolds, J. F.

See Carey, W. N. Jr., Shook, J. F. and Reynolds, J. F.

Rhodes, J. M.

See Hall, R. C., and Rhodes, J. M.

Rhodes, R. H.

See Nelson, R. C., and Rhodes, R. H.

Rice, W. L. R.

See Campbell, J. E. and Rice, W. L. R.
See Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

Richards, H. T.

See Hanson, M. P., Stickley, W. G., and Richards, H. T.

Riehl, W. A.

See Lucas, W. R., and Riehl, W. A.

Ritchie, T. and Plewes, W. G.

Moisture Penetration of Brick Masonry Panels, Published in ASTM BULLETIN, No. 249, Oct., 39 (TP 183).

Ritz, V. H. and Attix, F. H.

A Solid State Bragg-Gray Cavity Chamber—Summary of Proceedings of *STP 286*, 1203.

Roberts, A. G.

Improved NBS Abrasive Jet Method for Measuring Abrasion Resistance of Coatings, Published in ASTM BULLETIN, No. 244, Feb., 48 (TP 52).

Roberts, G. A.

Discussion, 778.

Robinson, W. M.

An Alignment Block and Clamp for Capillary Viscometers, Published in ASTM BULLETIN, No. 245, April, 69 (TP 75). (Technical Note).
A Reverse-Flow Viscometer, Published in ASTM BULLETIN, No. 246, May, 40 (TP 100). (Technical Note).

Rogers, E. D.

See Mertens, E. W., Coyne, L. D., and Rogers, E. D.

Romanko, John

Reactor Spectra Considerations in Radiation Effects Prediction—Summary of Proceedings of *STP 286*, 1203.

Rosenberg, S. J.

Discussion, 644, 687.

Rossin, A. D.

Radiation Damage in Steel: Considerations Involving the Effect of Neutron Spectra—Summary of Proceedings of *STP 286*, 1203.

Rothstein, P. and Turner, F. R.

Metallographic Specimen Preparation by Vibratory Polishing—Summary of Proceedings of *STP 285*, 1003.

Royen, M.

A Study of the Accelerated Aging of Vinyl Plastic Compounds in Modified Testing Oven, Published in ASTM BULLETIN, No. 243, Jan., 43 (TP 1).

Rudin, A.

See Birks, A. M., and Rudin, A.

Rudin, A., and Birks, A. M.

Measurement of Environmental Stress-Cracking of Polyethylene, Published in ASTM BULLETIN, No. 245, April, 60 (TP 66).

Ruprecht, W. J.

See March, J. L., Ruprecht, W. J., and Reed, G.

S

Sachs, G.

See Weiss, Volker, Schaeffer, G. T., Saule, A., and Sachs, George

Sachs, G., Gerberich, W. W., Weiss, V., and Latorre, J. V.

Low-Cycle Fatigue of Pressure-Vessel Materials, 512.

Sachs, G. and Sessler, J. G.

Effect of Stress Concentration on Tensile Strength of Titanium and Steel Alloy Sheet at Various Temperatures—Summary of Proceedings of *STP 287*, 1004.

Sankey, G. O.

Spin-Fracture Tests of Nickel-Molybdenum-Vanadium Rotor Steels, in the Brittle Fracture Range, 721 (*R*). Disc., 733.

Saule, A.

See Weiss, Volker, Shaeffer, G. T., Saule, A., and Sachs, George.

Schaeffer, G. T.

See Weiss, Volker, Schaeffer, G. T., Saule, A., and Sachs, George

Schjeldrup, H. C.

Prediction of Acoustic Fatigue Life—Summary of Proceedings of *STP 284*, 1001.

Schjodt, R.

Measurements of Human Reaction to Hardness of Floor Covering, Published in ASTM BULLETIN, No. 247, July, 53 (TP 119) (*R*).

Schmid, W. E.

Discussion, 1132, 1149.

Schmidt, R. J.

See Ellis, W. H. and Schmidt, R. J.

Schulte, C. A.

Predicting Creep Deflections of Plastic Beams, 895.

Schwartzbart, Harry

See Lehrer, William M., and Schwartzbart, Harry.

Schwebel, Abraham

Panel Consultant for Symposium on Nuclear Methods of Measuring Soil Density—Summary of Proceedings of *STP 293*, 1166.

Scott, A.

Urges Larger U. S. Effort in International Standardization, Published in ASTM BULLETIN, No. 249, Oct., 10.

Scoville, W., Jr.

See Boor, L., Hanok, M., Conant, F., and Scoville, W., Jr.

Seltzer, D. D.

Discussion, 971.

Sereda, P. J.

Measurement of Surface Moisture and Sulfur Dioxide Activity at Corrosion Sites, Published in ASTM BULLETIN, No. 246, May, 47 (TP 107).

Sessler, J. G.

See Sachs, George and Sessler, J. G.

Sessoms, O. V. P.

See Hale, D., Johnson, D. R., Dec, S. M., Coss, J. R., Sessoms, O. V. P., Hemmig, P. B., Rice, W. L. R., and Brocklehurst, R. E.

Shane, R. S.

On the Lap-Joint Shear Test for Metal-to-Metal Bonds—Summary of Proceedings of *STP 289*, 1002.

Preparation of Metal Surfaces for Adhesive Bonding—Summary of Proceedings of *STP 289*, 1002.

Shannon, J. L., Jr., Epsey, G. B., Repko, A. J. and Brown, W. F. Jr.

Effect of Carbon Content and Melting Practice on Room-Temperature Sharp-Edge-Notch Tensile Characteristics of H-11 Modified and 300M Sheet Steels, 761 (*R*). Disc., 778.

Sheehan, J. P.

Discussion, 787.

Shobert, E. I., II

Improving Secondary Education in Science (ACR notes), Published in ASTM BULLETIN, No. 246, May, 7.

Shook, J. F.

See Carey, W. N., Jr., Shook, J. F. and Reynolds, J. F.

Shoudy, A. A.

See Silliman, M. A., Shoudy, A. A., Huebotter, P. R., and Blessing, W. G.

Shuttleworth, C. G.

Discussion, Published in ASTM BULLETIN, No. 243, Jan., 51 (TP 9).

Silliman, M. A., Shoudy, A. A., Huebotter, P. R., and Blessing, W. G.

Irradiation Testing of Enrico Fermi Prototype Fuel Pins in the CP-5 Reactor—Summary of Proceedings of *STP 286*, 1203.

Simmons, Ward and Metzger, M. C.

Compilations of Chemical Composition and Rupture Strengths of Super-Strength Alloys, *STP 170-B*.

Simon, R. C.

Water Permeability Studies on Latex and Latex Paint Films, Published in ASTM BULLETIN, No. 249, Oct., 48 (TP 192).

Sinclair, G. M.

See Brock, G. W., and Sinclair, G. M.
See Stephens, R. I. and Sinclair, G. M.

Sinclair, G. M. and Feltner, C. E.

Fatigue Strength of Crystalline Solids—Summary of Proceedings of *STP 283*, 499.

Sinnott, M. J.

The Influence of Surfaces on the Properties of Materials—Summary of Proceedings of *STP 283*, 499.

Sisco, F. T.

The Pendulum Swings On (ACR notes), Published in ASTM BULLETIN, No. 248, Sept., 18.

Smith, C. R.

Design Applications for Improving Fatigue Resistance of Airplane Structures, 589. Disc., 601.

Smith, W. J.

The Labor of Technical Writing (ACR notes), Published in ASTM BULLETIN, No. 250, Dec., 12.

Smotryck, S. W.

See Bazett, D. J., and Smotryck, S. W.

Snow, R. B.

Techniques for Controlling Surface Pitting in Polishing Ceramic Materials—Summary of Proceedings of *STP 285*, 1003.

Soldan, H. M.

Discussion, 686.

Speight, F. Y.

Materials Research for Defense, Published in ASTM BULLETIN, No. 250, Dec., 6.

Spengeman, W. T. and Wormald, G.

Lightfastness Testing of Pigment Colors, Published in ASTM BULLETIN, No. 249, Oct., 29 (TP 173).

Squires, F. H.

From Certainty to Statistics, Published in ASTM BULLETIN, No. 243, Jan., 27.

Steele, L. E.

See Hawthorne, J. R. and Steele, L. E.

Steele, L. E. and Hawthorne, J. R.

Neutron Dosimetry for Materials Irradiation Studies—Summary of Proceedings of *STP 286*, 1203.

Stehsel, M. S.

See Waller, C. E. and Stehsel, M. S.

Steidel, R. F., Jr., and Makerov, C. E.

The Tensile Properties of Some Engineering Materials at Moderate Rates of Strain, Published in ASTM BULLETIN, No. 247, July, 57 (TP 123).

Steigerwald, E. A.

Delayed Failure of High-Strength Steel in Liquid Environments, 750.

Stengel, N. C.

Discussion, 811.

Stephens, R. I. and Sinclair, G. M.

A High-Temperature Vacuum, Axial Fatigue Testing Machine, Published in ASTM BULLETIN, No. 249, Oct., 44 (TP 188).

Stickley, G. W.

Additional Studies of Effects of Anodic Coatings on the Fatigue Strength of Aluminum Alloys, 577. Disc., 588.
See Hanson, M. P., Stickley, G. W., and Richards, H. T.

Stulen, F. B.

Discussion, 509.

Swayze, Myron A.

Discussion, 1018.

T

Taft, B. L.

See Belton, J. H., Godby, J. B., and Taft, B. L.

Terry, Milton

On the Analysis of Planned Experiments, Summary of Proceedings of the Symposium on Quality of Observations. Published in *Materials Research & Standards* (formerly ASTM BULLETIN), Vol. 1, No. 4, April, 1960, 263. (*See also*, 1204.)

Tiner, N. A. and Fischler, J. E.

A Study of Mechanical Behavior of Inconel 718 Alloy for Design of a Glide Vehicle, 825 (*R*).

Toolin, Parks

Discussion, 588.

Towns, R. L.

See Binder, D., Bopp, C. D. and Towns, R. L.

Trapp, W. J. and Lazan, B. J.

Material Properties that Affect Acoustical Fatigue Life and the Role of Damping—Summary of Proceedings of *STP 284*, 1001.

Turner, F. R.

See Rothstein, P. and Turner, F. R.

Tweed, P. B.

See Noonan, J. P., Heinemann, R. W., Lowell, S. J., and Tweed, P. B.

V

Van Zelst, T. W.

See Neville, O. K. and Van Zelst, T. W.

Vater, Eugene J.

See Hamaker, John C., Jr., and Vater, Eugene J.

Verbeck, George and Landgren, Robert

Influence of Physical Characteristics of Aggregates on Frost Resistance of Concrete, 1063 (*R*).

Voorhees, Howard R. and Freeman, James W.

The Elevated-Temperature Properties of Aluminum and Magnesium Alloys. (*Issued as a separate pamphlet STP 291.*)

W

Wallace, G. B.

See Higginson, E. C., and Wallace, G. B.

Waller, C. E. and Stehsel, M. L.

A Technique for Torsional Testing of Refractory Materials at Temperatures up to 2800 C—Summary of Proceedings of *STP 289*, 1002.

Waters, E. H., Hosking, J. S., and Hueber, H. V.

Tests for Potential and Past Moisture Expansion of Ceramic Building Units, Published in ASTM BULLETIN, No. 245, April, 55 (TP 61) (*R*).

Watson, J. F.

Introduction, Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials—Summary of Proceedings of *STP 297*, 1004.

Watson, J. F. and Christian, J. L.

Low-Temperature Properties of Cold-Rolled AISI Types 301, 302, 304 ELC and 310 Stainless Steel Sheet—Summary of Proceedings of *STP 287*, 1004.

Mechanical Properties of High Strength 301 Stainless Steel Sheet at 70, −320, and −423 F in the Base Metal and Welded Joint Configuration—Summary of Proceedings of *STP 287*, 1004.

Weaver, T. L.

See Michaels, A. I., Weaver, T. L., and Nelson, R. C.

Weibull, Waloddi

The Fatigue Damaging Effect of a Random Load—Summary of Proceedings of *STP 284*, 1001.

Weigel, J. F.

Investigation of the Fine and Coarse Aggregates of Northern Illinois for Alkali Reactivity, 1036.

Weiss, Volker

See Sachs, G., Gerberich, W. W., Weiss, V., and Latorre, J. V.

Weiss, Volker, Schaeffer, ~~G. T.,~~ Saule, A., and Sachs, George

Thermal Cycling Under Constant Load to Low Temperatures of Aluminum and Magnesium Alloys—Summary of Proceedings of *STP 287*, 1004.

Weissmann, G. F.

Discussion, 1149.

Wessel, E. T.

Brittle Fracture Strength of Metals—Summary of Proceedings of *STP 283*, 499.

Westbrook, J. H.

An Improved Microhardness Tester for High-Temperature Use, Published in ASTM BULLETIN, No. 246, May, 53 (TP 113).

White, M.

See Dorion, G. H., Burkhard, H., and White, M.

Whitehurst, E. A.

Introduction, Papers on Road and Paving Materials (Bituminous)—Summary of Proceedings of *STP 294*, 1167.

Wilder, A. B.

Discussions, 687, 893 1199.

Wilkov, Melvin A.

New Observations Relating to the Mechanism of Fatigue Failure, 540 (*R*). Disc., 559.

Willard, M.

Determination of the Thermal Stability of Disiloxane-Base Hydraulic Fluids, Published in ASTM BULLETIN, No. 249, Oct., 52 (TP 196).

Williams, F. S., Beck, W. and Janowsky, E. J.

A Notched Ring Specimen for Hydrogen Embrittlement Studies, 1192. Disc., 1199.

Winne, D. H.

Discussion, 733.

Winniford, R. S.

The Influence of Asphalt Composition on Its Rheology—Summary of Proceedings of *STP 294*, 1167.

Wood, Lyman W., Erickson, E. C. O., and Dohr, A. W.

Strength and Related Properties of Wood Poles. (*Issued as a separate pamphlet STP 295.*)

Woolf, D. O.

Discussion, Published in ASTM BULLETIN, No. 243, Jan., 68 (TP 26).

Woolley, R. H.

See Jenike, A. W., Elsey, P. J., and Woolley, R. H.

Wormald, G.

See Spengemen, W. T. and Wormald, G.

Wyman, L. L.

Summary, Symposium on Methods of Metallographic Specimen Preparation—Summary of Proceedings of *STP 285*, 1003.

Y

Yen, C. S.

Stress Distribution in Single-Shear Sheet-Specimens—Summary of Proceedings of *STP 289*, 1002.

Youden, W. J.

Some Observations on Evaluating Accuracy—Summary of Proceedings of the Symposium on Quality of Observations. Published in *Materials Research & Standards* (formerly ASTM BULLETIN), Vol. 1, No. 4, April, 1961, 263. (*See also*, 1204.)

Young, J. F.

Metallurgy for Non-Metallurgical Engineers? Published in ASTM BULLETIN, No. 244, Feb., 15.

Youngquist, W. G. and Kuenzi, E. W.

Shear and Torsion Testing of Wood, Plywood, and Sandwich Constructions at the U. S. Forest Products Laboratory—Summary of Proceedings of *STP 289*, 1002.

Z

Zandman, Felix

Maximum Shear Strain Measurements and Determination of Initial Yielding by the Use of the Photoelastic Coating Technique—Summary of Proceedings of *STP 289*, 1002.

Zapel, E. J.

Discussion, 601.

The Design and Development of a Tensile-Loaded Shear Specimen—Summary of Proceedings of *STP 289*, 1002.

Zaretsky, E. V. and Anderson, W. J.

Relation Between Rolling-Contact Fatigue Life and Mechanical Properties for Several Aircraft Bearing Steels, 627 (*R*). Disc., 643.

Zegarra, E. J.

Observed Settlement of Preloaded Soft, Fine-Grained Soils, 1109. Disc., 1118.

Zoldners, N. G.

Effect of High Temperatures on Concretes Incorporating Different Aggregates, 1087 (*R*).

SUBJECT INDEX

1960 Technical Papers and Committee Reports

A

- Abrasion
  - resistance of coatings, method for measuring (Roberts). Published in ASTM Bulletin, No. 244, Feb., 48 (TP 52).
  - resistance of plastics, testing (Boor). Published in ASTM Bulletin, No. 244, Feb., 43 (TP 47).
- Absorption Spectroscopy
  - Report of Committee E-13, 482
- Accuracy
  - what price test methods (McPherson). Published in ASTM Bulletin, No. 244, Feb. 7. (ACR notes).
- Acoustical Materials
  - Report of Committee C-20, 289.
  - Report of Committee D-14, 371.
- ACR Notes
  - improving secondary education in science (Schobert). Published in ASTM Bulletin, No. 246, May, 7.
  - meaning of measurement (Baker). Published in ASTM Bulletin, No. 245, April, 42.
  - published in ASTM Bulletin, No. 244, Feb., 7; No. 245, April, 42; No. 246, May, 7; No. 248, Sept., 18; No. 250, Dec., 12.
  - the labor of technical writing (Smith). Published in ASTM Bulletin, No. 250, Dec., 12.
  - the pendulum swings on (metallurgy) (Sisco). Published in ASTM Bulletin, No. 248, Sept., 18.
  - what price accurate test methods (McPherson). Published in ASTM Bulletin, No. 244, Feb., 7.
- Adhesives
  - portable testing device for (O'Kelly). Published in ASTM Bulletin, No. 250, Dec. 32 (TP 210).
  - Report of Committee D-14, 371.
- Aggregates
  - alkali reactivity (Weigel), 1036.
  - concrete, separation of lightweight material from (Higginson and Wallace). Published in ASTM Bulletin, No. 243, Jan., 60 (TP 18).
  - effect of physical characteristics on frost resistance of concrete (Verbeck & Landgren), 1063 (*R*).
  - low density
    - effects of early freezing on concrete (Benjamin and Ratliff), 1080.
  - Report of Committee C-9, 266.
- Aging
  - accelerated, of vinyl plastic compounds (Royen). Published in ASTM Bulletin, No. 243, Jan., 43 (TP 1).
- Aircraft
  - bearing steels
    - rolling contact fatigue life and mechanical properties (Zaretsky & Anderson), 627 (*R*). Disc., 643.
  - *See* Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials—Summary of Proceedings of *STP 287*, 1004.
- Aircraft Materials
  - Symposium on Low Temperature Properties—Summary of Proceedings of *STP 287*, 1004.
- Air-Entrainment
  - of standard Ottawa sand, test method for (DeFore and Corah). Published in ASTM Bulletin, No. 248, Sept., 48 (TP 162).
- Airplane structures
  - design application
    - improving fatigue resistance (Smith), 589, Disc., 601.
- Alkali Reactivity
  - aggregate (Weigel), 1036.
- Alloy Steel
  - Report of Committee A-6, 124.
- Alloying Additions
  - effects on hot-cracking of austenitic
    - chromium-nickel stainless steels (Hull), 667 (*R*). Disc., 686
- Alloys
  - high-temperature
    - Report of Joint Committee on Effect of Temperature on the Properties of Metals, 241.
  - super-strength
    - chemical composition and rupture strengths (Simmons and Metzger) (*issued as STP 170-B*, 1960).
- Aluminum
  - alloys
    - atmospheric exposure (Adams and Dougherty). Report of Committee B-7 (appendix II), 186.
    - elevated temperature properties (Voorhees and Freeman) (*issued as STP 291*).
    - exposure tests of die-cast test bars. Report of Committee B-6 (appendix II), 165.
    - fatigue strength of
      - effects of anodic coatings (Stickley), 577 Disc., 588.

- anodic coatings
  - Report of Committee B-8, 204.
- atmospheric exposure tests
  - ASTM test sites (Adam and Dougherty). Report of Committee B-7 (appendix II), 186.
- corrosion of coated hardware
  - Report of Subcommittee XVI of Committee A-5, 116.
- panel discussion on surface appearance—Summary of Proceedings, 1205.
- Report of Committee B-1, 130.
- Report of Committee B-6, 160.

Analysis, Chemical
- Report of Committee E-15, 486.

Annual Meeting
- Report of Board of Directors, 45.
- Summary of Proceedings of Sixty-third Annual Meeting, 1.
- *See also* Meetings.

Anodic coatings
- aluminum alloys
  - effect on fatigue strength (Stickley), 577. Disc., 588.

Antifreezes
- *See* Engine Antifreezes.

Appearance
- panel discussion on aluminum surfaces—Summary of Proceedings, 1205.
- Report of Committee E-12, 480.

Aromatic Hydrocarbons
- interlaboratory testing of
  - proposed recommendations for, 381.
- Report of Committee D-16, 377.

Asbestos-Cement Products
- Report of Committee C-17, 284.

Askarels
- Report of Committee D-27, 429.

Asphalt
- coating grade, predicting weatherability from asphaltene characteristics (Mertens). Published in ASTM BULLETIN, No. 250, Dec. 40 (TP 218).
- Report of Committee D-4, 328.
- Report of Committee D-8, 345.
- *See also* Session on Road and Paving Materials —Summary of Proceedings of *STP 294*, 1167.

ASTM
- fellowship program, grants, 54. Published in ASTM BULLETIN, No. 244, Feb., 14; No. 245, April, 37.
- finances—1959. Published in ASTM BULLETIN, No. 244, Feb., 8.
- headquarters staff
  - Marshall, T. A., named new executive secretary. Published in ASTM BULLETIN, No. 249, Oct., 5.
  - Painter, R. J., given sick leave. Published in ASTM BULLETIN, No. 244, Feb., 14; No. 245, April, 35.
- publications 1960–1961. Published in ASTM BULLETIN, No. 249, Oct., 12.
- review of the year 1959. Published in ASTM BULLETIN, No. 243, Jan., 5.
- role in occupational safety (Mochel). Published in ASTM BULLETIN, No. 246, May, 13.

Atmospheric Corrosion
- Report of Committee B-7, 170.
- twenty years
  - zinc-coated and uncoated wire (Reinhart) (*issued as STP 290*).

Atmospheric Exposure
- aluminum and magnesium alloys
  - ASTM test sites (Adam and Dougherty). Report of Committee B-7 (appendix II), 186.
- hardware, 29-yr report
  - Report of Subcommittee XVI of Committee A-5, 116.
- Report of Advisory Committee on Corrosion, 234.
- Report of Committee A-5, 111.

Atmospheric Sampling and Analysis
- Report of Committee D-22, 417.

Atmospheric Testing
- punch card handling of data (Ailor and Hodgson). Published in ASTM BULLETIN, No. 250, Dec., 45 (TP 223).

Awards
- citations for quality of presentation, 1959. Published in ASTM BULLETIN, No. 247, July, 15.
- committee D-12, to A. M. Schwartz. Published in ASTM BULLETIN, No. 245, April, 41.
- Harold De Witt Smith, to W. von Bergen. Published in ASTM BULLETIN, No. 245, April, 46.
- honorary membership. Published in ASTM BULLETIN, No. 247, July, 21.
- medals and awards. Published in ASTM BULLETIN No. 247, July 28.
  - Charles B. Dudley Medal. Published in ASTM BULLETIN No. 247, July, 28.
  - Max Hecht Award. Published in ASTM BULLETIN No. 247, July, 28.
  - C. A. Hogentogler Award. Published in ASTM BULLETIN No. 247, July, 28.
  - Frank E. Richart Award. Published in ASTM BULLETIN No. 247, July, 29.
  - Richard L. Templin Award. Published in ASTM BULLETIN No. 247, July, 29.
  - Sanford E. Thompson Award. Published in ASTM BULLETIN No. 247, July, 30.
  - Sam Tour Award. Published in ASTM BULLETIN No. 247, July, 29.
- SES-ASTM, 1959. Published in ASTM BULLETIN, No. 243, Jan., 42.
- Report of the Board of Directors, 50.

B

Barium Oxide
- in portland cement, gravimetric method for determination of (Ford). Published in ASTM BULLETIN, No. 247, July, 77 (TP 143).

Barriers
- flexible
  - Report of Committee F-2, 495.

- Basic Refractory Brick
  - Report of Committee C-8, 260.
- Beryllium
  - Report of Committee B-2, 135.
  - structural (Muvdi), 812 (*R*). Disc., 823.
- Bituminous Materials
  - Report of Committee D-4, 328.
  - Report of Committee D-8, 345.
- Bituminous Paving Materials
  - Report of Committee D-4, 328.
  - *See also* Session on Road and Paving Materials—Summary of Proceedings of *STP 294*, 1167.
- Bituminous Roofing Materials
  - Report of Committee D-8, 345.
- Board of Directors
  - Auditor's report (appendix I), 57.
  - Report of Administrative Committee on District Activities (appendix V), 84.
  - Report of Administrative Committee on Education in Materials (appendix VII), 86.
  - Report of Administrative Committee on Papers and Publications (appendix IV), 79.
  - Report of Administrative Committee on Research (appendix III), 78.
  - Report of Administrative Committee on Simulated Service Testing (appendix VI), 85.
  - Report of Administrative Committee on Standards (appendix II), 71.
  - Report of Board of Directors, 40.
- Boiler Feedwater
  - Report of Committee D-19, 393.
- Brass
  - Report of Committee B-5, 146.
- Brick
  - panels, moisture penetration of (Ritchie and Plewes). Published in ASTM BULLETIN, No. 249, Oct., 39 (TP 183).
  - Report of Committee C-15, 277.
  - *See also* Fireclay Brick *and* Basic Refractory Brick.
- Brittleness Temperature
  - of polyethylene, measurement of (Birks and Rudin). Published in ASTM BULLETIN, No. 246, May, 49 (TP 109).
  - of polyethylene, test for (Bata and Ito). Published in ASTM BULLETIN, No. 248, Sept., 55 (TP 165) (*Technical Note*).
- Bronze
  - Report of Committee B-5, 146.
  - tension of phosphor bronze rod, alloy A (Gohn and Bader), 843.
- Brushes
  - carbon, technique for measuring wear in an atmosphere containing silicone vapor (Axelson and Precopio). Published in ASTM BULLETIN, No. 244, Feb., 40 (TP 44).
- Building Constructions
  - Report of Committee E-6, 468.
- Building Stone
  - Report of Committee C-18, 288.
- Bulk Solids
  - flow properties (Jenike, Elsey, and Woolley), 1168. Disc. 1182.

C

- Cable
  - insulating
    - Report of Committee D-11, 355.
  - Report of Committee B-1, 130.
- Carbon Black
  - Report of Committee D-24, 423.
- Casein
  - Report of Committee D-25, 426.
- Cast Iron
  - Report of Committee A-3, 108.
- Cathodes
  - Report of Committee F-1, 491.
- Cellulose
  - Report of Committee D-23, 420.
- Cellulose Viscosity
  - Report of Committee D-23, 420.
- Cement
  - flexures and compression tests (Kaplan), 1006 (*R*). Disc., 1018.
  - portland
    - flame photometric method to determine magnesium, manganic, sodium, and potassium oxides in (Ford). Published in ASTM BULLETIN, No. 250, Dec. 25 (TP 203).
    - gravimetric method for determination of barium oxide in (Ford). Published in ASTM BULLETIN, No. 247, July, 77 (TP 143).
    - gravimetric determination of strontium oxide in (Ford). Published in ASTM BULLETIN, No. 245, April, 71 (TP 77).
    - movement of sodium in (Hall and Rhodes). Published in ASTM BULLETIN, No. 245, April, 66 (TP 72).
  - portland pastes, hydrated
    - X-ray diffraction investigation (Kantro, Copeland, and Anderson), 1020 (*R*). Disc., 1035.
  - Report of Committee C-1, 247.
  - thermal insulating
    - Report of Committee C-16, 281.
- Cement Reference Laboratory
  - Report of Committee C-1, 247.
- Ceramic
  - building units, potential and past moisture expansion, tests for (Waters, Hosking, and Hueber). Published in ASTM BULLETIN, No. 245, April, 55 (TP 61).
  - Report of Committee C-22, 295.
  - Report of Committee F-1, 491.
- Ceramic Coatings
  - Report of Committee C-22, 295.
- Ceramic Whitewares
  - Report of Committee C-21, 291.
- Chemical Analysis
  - Report of Committee E-2, 448.
  - Report of Committee E-3, 452.
- Chemicals
  - industrial analysis and testing
    - Report of Committee E-15, 486.
  - Report of Committee D-26, 428.
  - rubber
    - Report of Committee D-24, 423.

Chemical-Resistant Mortars
  Report of Committee C-15, 277.
Chemical-Resistant Units
  Report of Committee C-15, 277.
Chlorinated Solvents
  Report of Committee D-26, 428.
Classification
  of elastomeric vulcanizates, method for (Catton, Edwards, and Loring). Published in ASTM BULLETIN, No. 243, Jan., 69 (TP 27).
Clay
  remolded Niagara
    compressive strength (Bazett and Smotrych), 1124. Disc., 1132.
Clay Pipe
  Report of Committee C-4, 255.
Clay Tile
  Report of Committee C-15, 277.
Coal
  Report of Committee D-5, 332.
Coal Chemicals
  Report of Committee D-16, 377.
Coatings
  abrasion resistance of, method for measuring (Roberts). Published in ASTM BULLETIN, No. 244, Feb., 48 (TP 52).
  aluminum, anodic
    Report of Committee B-8, 204.
  electrodeposited
    Report of Committee B-8, 204.
    Report of Subcommittee III of Committee B-8. Part I: Thickness Determination of Electrodeposits, 207. Part II: Evaluation of Measuring Devices for Thickness of Zinc Electrodeposited on Steel, 218.
Coke
  Report of Committee D-5, 332.
Color
  Report of Committee E-12, 480.
Columbium
  Report of Committee B-2, 135.
Combustion Characteristics
  of diesel fuel, use of 2,2,4,4,6,8,8-heptamethylnonane as primary reference (Mears, David, and Howard). Published in ASTM BULLETIN, No. 245, April, 75 (TP 81).
Committees
  in defense of (Burns), No. 246, May, 24.
  new officers for ASTM technical. Published in ASTM BULLETIN, No. 248, Sept., 20.
Compression
  determining strength of concrete (Kaplan), 1006 (*R*). Disc., 1018.
  remolded Niagara clay (Bazett and Smotrych), 1124. Disc., 1132.
  sheet magnesium
    utilizing rapid heating (Fenn), 940 (*R*). Disc., 955.
Concrete
  aggregate, separation of lightweight material from (Higginson and Wallace). Published in ASTM BULLETIN, No. 243, Jan., 60 (TP 18).
  effect of high temperature (Zoldners), 1087 (*R*).
  frost resistance
    influence of physical characteristics of aggregates (Verbeck & Landgren), 1063 (*R*).
  low-density aggregate type
    effects of early freezing (Benjamin and Ratliff), 1080.
  Report of Committee C-9, 266.
Concrete Aggregates
  Report of Committee C-9, 266.
Concrete Masonry Units
  Report of Committee C-15, 277.
Concrete Mixers
  performance tests (Narrow), 1047. Disc., 1061.
Concrete Pipe
  Report of Committee C-13, 275.
Conductors
  Report of Committee B-1, 130.
Contact Materials, Electrical
  Report of Committee B-4, 143.
Containers
  Report of Committee D-10, 353.
Cooperative Activities
  Report of Board of Directors, 54.
Copper
  annealed
    fatigue notch sensitivity (Mann), 602 (*R*).
  copper-base casting alloy
    Mechanical and physical properties (Johnson & Kura), 796. Disc., 811.
  Report of Committee B-2, 135.
  Report of Committee B-5, 146.
  See also Proposed Recommendations.
Copper Alloys
  properties at elevated temperatures (Crowe). Published in ASTM BULLETIN, No. 250, Dec. 30 (TP 208).
Copper Wire
  Report of Committee B-1, 130.
Corrosion
  atmospheric
    low alloy steels (Copson), 650 (*R*). Disc., 666.
  Report of Advisory Committee on Corrosion, 234.
  Report of Committee A-5, 111.
  Report of Committee A-10, 126.
  Report of Committee B-3, 141.
  Report of Committee B-6, 160.
  Report of Committee B-8, 204.
  sulfur dioxide activity and surface moisture at corrosion sites, measurement of (Sereda). Published in ASTM BULLETIN, No. 246, May, 47 (TP 107).
  tests on hardware. Report of Subcommittee XVI of Committee A-5, 116.
  twenty-year atmospheric
    zinc-coated and uncoated wire (Reinhart) (*issued as STP 290*).
Corrosion-Resisting Steel
  Report of Committee A-10, 126.
Cracking
  of polyethylene under stress, environmental, measurement of (Rudin and Birks). Published in ASTM BULLETIN, No. 245, April, 60 (TP 66).

Creep
  anelastic, effect of temperature interruption on (Disc.). Published in ASTM BULLETIN, No. 243, Jan., 54 (TP 12).
  elastomers, rheological testing of, at low temperatures (Boor, Hanok, Conant, and Scoville). Published in ASTM BULLETIN, No. 246, May, 25 (TP 85).
  properties of compression-molded polyethylene (Gohn and Cummings). Published in ASTM BULLETIN, No. 247, July, 64 (TP 130).
Creep Deflections
  plastic beams (Schulte), 895.
Creep-Rupture
  carbon and low alloy steel at 1200 to 1800 F (Randall), 885. Disc., 893.
Crystalline Solids
  Symposium on Nature and Origin of Strength of Materials, Summary of Proceedings of *STP 283*, 499:
    brittle fracture strength (Wessel).
    ductile ceramic research (Parker).
    effects of size and shape on fracture (Irwin).
    fatigue strength (Sinclair and Feltner).
    influences of surfaces of materials (Sinnott).
    luncheon address
      national role of materials research (Baker).
    mechanical properties of semiconductors (Hobstetter).
    motions and yield strength of solids (Gilman).
    resistance to creep in metals and alloys (Garofalo).
    solid state theory (Brooks).

D

Data Centers
  are they the answer? Published in ASTM BULLETIN, No. 246, May, 14.
De Witt Smith Medal
  to W. von Bergen, 1960. Published in ASTM BULLETIN, No. 245, April, 46.
Defense
  materials research for (Speight). Published in ASTM BULLETIN, No. 250, Dec. 6.
Density
  relative, of sand, measurement of (Kitago and Kozaki). Published in ASTM BULLETIN, No. 248, Sept., 36 (TP 150).
Detergents
  Report of Committee D-12, 361.
Detonation
  impact, materials in contact with liquid oxygen, instrument for testing sensitivity to (Lucas and Riehl). Published in ASTM BULLETIN, No. 244, Feb., 29 (TP 33).
Die Casting
  Report of Committee B-6, 160.
Dielectric Strength
  determination of, insulating oil (Ast). Published in ASTM BULLETIN, No. 250, Dec., 36 (TP 214).
District Activities
  published in ASTM BULLETIN, No. 243, Jan., 42; No. 244, Feb., 16; No. 245, April, 43; No. 246, May, 19; No. 247, July, 39; No. 248, Sept., 19; No. 249, Oct., 23; No. 250, Dec., 16.
  Report of Administrative Committee on District Activities, 84.
  Report of Board of Directors, 46.
Dosimetry
  See Symposium on Radiation Effects and Radiation Dosimetry—Summary of Proceedings of *STP 286*, 1203.

E

Education
  secondary, improving science in (Schobert). Published in ASTM BULLETIN, No. 246, May, 7 (ACR notes).
Education in Materials
  Report of Administrative Committee on Education in Materials, 86.
Elastomers
  method for classifying (Catton, Edwards, and Loring). Published in ASTM BULLETIN, No. 243, Jan., 69 (TP 27).
  rheological testing of, at low temperatures (Boor, Hanok, Conant, and Scoville). Published in ASTM BULLETIN, No. 246, May, 25 (TP 85).
Electrical Contact
  bibliographical abstracts (*issued as STP 56-N*, 1959).
Electrical Contact Materials
  Report of Committee B-4, 143.
Electrical Heating Alloys
  Report of Committee B-4, 143.
Electrical Insulating Liquids and Gases
  Report of Committee D-27, 429.
Electrical Insulating Materials
  Report of Committee D-9, 347.
  Report of Committee D-20, 402.
Electrical Insulation
  Report of Committee F-1, 491.
Electrical Testing
  Report of Committee D-9, 347.
Electrodeposited Coatings
  Report of Committee B-8, 204.
  Report of Subcommittee III of Committee B-8. Part I: Thickness Determination of Electrodeposits, 207. Part II: Evaluation of Measuring Devices for Thickness of Zinc Electrodeposited on Steel, 218.
Electromagnetic Testing
  Report of Committee E-7, 471.
Electron Metallography
  Report of Committee E-4, 457.
Electron Tube Materials
  Report of Committee F-1, 491.
Elevated Temperature
  aluminum and magnesium alloys (Voorhees and Freeman) (*issued as STP 291*).
  application of induction heating to short-time tension testing (Levitt and Martin), 974. Disc., 984.
  correlating parameters
    stress-rupture (Clauss), 905 (*R*).

creep rupture of carbon and low alloy steel (Randall), 885. Disc., 893.
fatigue and tension
unalloyed arc-cast molybdium (Brock and Sinclair), 530. Disc., 538.
Inconel relaxation characteristics (Kennedy & Douglas), 867 (*R*). Disc., 881.
properties of copper alloys (Crowe). Published in ASTM BULLETIN, No. 250, Dec. 30 (TP 208).
Report of Joint ASTM-ASME Committee on Effect of Temperature on the Properties of Metals, 241.
Embrittlement
See Hydrogen Embrittlement.
Emission Spectroscopy
Report of Committee E-2, 448.
Enamel, Porcelain
Report of Committee C-22, 295.
Engine Antifreezes
Report of Committee D-15, 375.
Expansion
moisture, potential and past, of ceramic building units, tests for (Waters, Hosking, and Hueber). Published in ASTM BULLETIN, No. 245, April, 55 (TP 61).
Exposure Tests
aluminum alloy die-cast test bars. Report of Committee B-6 (appendix II), 165.
outdoor, Langley as a unit for timing (Caryl). Published in ASTM BULLETIN, No. 243, Jan., 55 (TP 13).

F

Fabrics
gripping of, during physical tests, technique for (Plitt and Dunlap). Published in ASTM BULLETIN, No. 246, May, 33 (TP 93).
Report of Committee D-13, 365.
See also Textiles.
Facing Tile
Report of Committee C-15, 277.
Fatigue
aircraft bearing steel
rolling contact and mechanical properties (Zaretsky & Anderson), 627 (*R*). Disc., 643.
airplane structures (Smith), 589. Disc., 601.
aluminum alloys
effects of anodic coatings (Stickley), 577, Disc., 588.
annealed copper
notch sensitivity (Mann), 602 (*R*).
elevated temperature
unalloyed arc-cast molybdium (Brock and Sinclair), 530. Disc., 538.
failure
mechanism of (Wilkov), 540 (*R*). Disc., 559.
high-strength steels (Frankel, Bennett & Carman), 501. Disc., 509.
low cycle
pressure vessel materials (Sachs, Gerberich, Weiss and Latorre), 512 (*R*).
metals subjected to triaxial stresses (Lehrer and Schwartzbart), 610 (*R*).
R. R. Moore rotating-beam
effect of specimen preparation (Gohn and Fox), 560 (*R*). Disc., 568.
Report of Committee E-9, 474.
Fatigue, Acoustical
Symposium. Summary of Proceedings of *STP 284*, 1001:
damaging effect of a random load (Weibull).
experimental techniques and equipment (Forney).
introduction (Lazan).
material properties and the role of damping (Trapp and Lazan).
prediction of life (Schjeldrup).
structural metals under random loading (Freudenthal).
Fatigue Testing
high-temperature, vacuum, axial, testing machine (Stephens and Sinclair). Published in ASTM BULLETIN, No. 249, Oct., 44 (TP 188).
Fibers
Report of Committee D-13, 365.
Filter Block, Clay
Report of Committee C-15, 277
Finances
ASTM, 1959, Published in ASTM BULLETIN, No. 244, Feb., 8.
auditor's report, 57.
Report of Board of Directors, 51.
Fire Testing
proposed method
for surface flammability of building materials using an 8-ft tunnel furnace. Report of Committee E-5 (appendix), 462.
Report of Committee E-5, 460.
Fireclay Brick
Report of Committee C-8, 260.
Flexible Barriers
Report of Committee F-2, 495.
Flexural Strength
phenolic moldings (Elmer and Harrington). Published in ASTM BULLETIN, No. 249, Oct., 35 (TP 179).
Flexure
determining strength of concrete (Kaplan), 1006 (*R*). Disc., 1018.
Floor Covering
hardness of, measurement of human reaction to (Schjodt). Published in ASTM BULLETIN, No. 247, July, 53 (TP 119).
Floor Polishes
Report of Committee D-21, 415.
Floor Wax
Report of Committee D-21, 415.
Flow
bulk solids (Jenike, Elsey, and Woolly), 1167. Disc., 1182.
Fluids, Hydraulic
disiloxane-base, thermal stability of (Willard). Published in ASTM BULLETIN, No. 249, Oct., 52 (TP 196).
Fracture Testing
sheet materials, high-strength (report of special ASTM committee). Published in ASTM BULLETIN, No. 243, Jan., 29; No. 244, Feb., 18.

Freezing rate
  importance in frost action in soils (Penner), 1151 (*R*).
Fuels
  diesel, use of 2,2,4,4,6,8,8-heptamethylnonane as primary reference (Mears, David, and Howard). Published in ASTM BULLETIN, No. 245, April, 75 (TP 81).
  gaseous
    Report of Committee D-3, 326.
  Report of Committee D-2, 316.

G

Gaseous Fuels
  Report of Committee D-3, 326.
Gasolines
  determination of tetraethyllead. Published in ASTM BULLETIN, No. 250, Dec., 13.
  Report of Committee D-2, 316.
  See also Petroleum *and* Olis.
Glass
  research at the National Bureau of Standards. Published in ASTM BULLETIN, No. 247, July, 45.
Glide vehicle
  mechanical behavior of Inconel 718 alloy (Tiner & Fischler), 825 (*R*).
Gloss
  Report of Committee E-12, 480.
Glycerin
  methods for phthalate ester color, historical review and supporting data. Report of Committee D-1 (appendix I), 311.
Granite
  building
    Report of Committee C-18, 288.
Grout
  chemical, permeability of sand stabilized with (Dorion, Burkhard, and White). Published in ASTM BULLETIN, No. 250, Dec. 34 (TP 212).
Gun Firing
  devices for simulating under, static conditions (Noonan, Heinemann, Lowell, and Tweed). Published in ASTM BULLETIN, No. 248, Sept., 33 (TP 147).
Gypsum
  Report of Committee C-11, 272.
Gypsum Plaster
  normal consistency of, method for determination (Kuntze). Published in ASTM BULLETIN, No. 246, May, 35, (TP 95).
  physical changes in setting (Disc.). Published in ASTM BULLETIN, No. 243, Jan., 51 (TP 9).

H

Hardness
  microhardness tester for high-temperature use (Westbrook). Published in ASTM BULLETIN, No. 246, May, 53 (TP 113).
  of floor coverings, measurement of human reaction to (Schjodt). Published in ASTM BULLETIN, No. 247, July, 53 (TP 119).
  of plastics, testing (Boor). Published in ASTM BULLETIN, No. 244, Feb., 43 (TP 47).
  polymeric materials, penetration tester for measuring (Gardiner, Jordan, and Adams). Published in ASTM BULLETIN, No. 246, May, 38 (TP 98).
  SAE 4340 steel, low-temperature, correlation with tensile strength (Nunes and Larson). Published in ASTM BULLETIN, No. 249, Oct. 25, (TP 169).
  testing
    Report of Committee E-1, 439.
Hardware
  Report of Committee A-5, 111.
  tests
    Report of Subcommittee XVI of Committee A-5, 116.
Heptamethylnonane
  use as a primary reference diesel fuel (Mears, David, and Howard). Published in ASTM BULLETIN, No. 245, April, 75 (TP 81).
High Temperature
  tester for microhardness (Westbrook). Published in ASTM BULLETIN, No. 246, May, 53 (TP 113).
  vacuum, axial fatigue testing machine (Stephens and Sinclair). Published in ASTM BULLETIN, No. 249, Oct., 44 (TP 188).
  vacuum equipment for treatment of reactive and refractory metals (Feild and Peckham). Published in ASTM BULLETIN, No. 244, Feb., 34 (TP 38).
Highway Materials
  Report of Committee D-4, 328.
Honorary Membership
  ASTM. Published in ASTM BULLETIN, No. 247, July, 21.
Honors and Awards
  Report of Board of Directors, 50.
Hot-cracking
  effect of alloying additions
    austenitic chromium-nickel stainless steels (Hull,) 667 (*R*). Disc., 686.
Hydraulic Cement
  Report of Committee C-1, 247.
Hydraulic Fluids
  disiloxane-base, thermal stability of (Willard). Published in ASTM BULLETIN, No. 249, Oct., 52 (TP 196).
Hydrocarbons
  aromatic
    interlaboratory testing, proposed recommendations for. Report of Committee D-16 (appendix), 381.
    Report of Committee D-16, 377.
Hydrogen Embrittlement
  notched ring specimens (Williams, Beck, and Jankowsky), 1192. Disc., 1199.

I

Ignition Characteristics
  of diesel fuel, use of 2,2,4,4,6,8,8-heptamethylnonane as primray reference (Mears, David, and Howard). Published in ASTM BULLETIN, No. 245, April, 75 (TP 81).
Impact
  phenolic moldings (Elmer and Harrington). Published in ASTM BULLETIN, No. 249, Oct. 35, (TP 179).

  - sensitivity, determining characteristics of materials with highly reactive oxidizers (Kopituk). Published in ASTM Bulletin, No. 250, Dec. 52 (TP 229).
  - sensitivity to detonation of materials in contact with liquid oxygen, instrument for testing (Lucas and Riehl). Publsihed in ASTM Bulletin, No. 244, Feb., 29 (TP 33).
  - tensile properties of metals (Steidel and Makerov). Published in ASTM Bulletin, No. 247, July, 57 (TP 123).
- Industrial Chemicals
  - analysis and testing
    - Report of Committee E-15, 486.
- Industrial Waste Water
  - Report of Committee D-19, 393.
- Inks
  - printing, and ink vehicles, modified MacMichael viscometer to determine consistency of (Cummings). Published in ASTM Bulletin, No. 250, Dec., 49 (TP 227).
- Insulating Materials
  - electrical
    - Report of Committee D-9, 347.
  - thermal
    - Report of Committee C-16, 281.
- Insulating Wire and Cable
  - Report of Committee D-11, 355.
- Insulation
  - magnet wire, compatibility with epoxy encapsulating resins (Lee). Published in ASTM Bulletin, No. 247, July, 69 (TP 135).
  - oils, insulating, development of (Furby and Hanly). Published in ASTM Bulletin, No. 248, Sept., 41 (TP 155).
- International Relations
  - Report of Administrative Committee on Standards, 71.
- Iron
  - cast
    - Report of Committee A-3, 108.
  - corrosion
    - Report of Advisory Committee on Corrosion, 234.
    - Report of Committee A-5, 111.
  - Report of Committee E-3, 452.

J

- Joint Sealants
  - Report of Committee C-24, 298.

L

- Laboratories
  - plan for the self-qualification of (McPherson). Published in ASTM Bulletin, No. 246, May, 17.
- Lamination Factor
  - of small steel disk, correlation with roughness factor (Begovich). Published in ASTM Bulletin, No. 248, Sept., 56 (TP 166).
- Langley
  - unit for timing outdoor exposures (Caryl), Published in ASTM Bulletin, No. 243, Jan. 55 (TP 13).
- Latex
  - paints, permeability to water (Simon). Published in ASTM Bulletin, No. 249, Oct., 48 (TP 192).
- Lead
  - corrosion of coated hardware
    - Report of Subcommittee XVI of Committee A-5, 116.
  - Report of Committee B-2, 135.
- Leather
  - Report of Joint Committee on Leather, 435.
- Lectures
  - Gillett (8th), nuclear fuel element development (Dalzell). Published in ASTM Bulletin, No. 245, April, 10. (*also issued as a separate pamphlet*).
  - Marburg (34th), solar energy (Daniels). Published in ASTM Bulletin, No. 245, April, 10. (*also issued as a separate pamphlet*).
- Light Metals
  - Report of Committee B-7, 170.
  - *See also* Proposed Recommendations.
- Lightfastness
  - of pigment colors, testing of (Spengeman and Wormald). Published in ASTM Bulletin, No. 249, Oct., 29 (TP 173).
- Lime
  - Report of Committee C-7, 257.
- Lithium
  - Report of Committee B-2, 135.
- Long-Range Planning
  - Report of Board of Directors, 56.
- Low-Temperature
  - Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials—Summary of Proceedings of *STP 287*, 1004:
    - aluminum alloy weldments (Lucas and Cataldo).
    - aluminum and magnesium alloys theraml cycling (Weiss, Schaeffer, Saul, and Sachs).
    - aluminum sheet alloys—sharp-notch behavior (Hanson, Stickley, and Richards).
    - cold rolled AISI types 301, 302, 304 ELC and 310 stainless steel (Watson and Christian).
    - effect of rolling on stainless steel (Mayne)
    - 43XX-Vanadium modified and 5% Cr sheet steels—effect of carbon content (Klier).
    - magnesium alloys (Fenn).
      - mechanical properties (Reed, Mikesell, and Greeson).
    - materials for use at liquid hydrogen temperatures (Belton, Godby and Taft).
    - steel
      - mechanical properties in base metal and welded joint configuration (Watson and Christian).
      - stainless and low-alloy steels (Campbell and Rice).
    - titanium and steel sheet alloys
      - effect of stress concentration on tensile strength (Sachs and Sessle).
    - titanium sheet alloys—sharp-edge-notch tensile characteristics (Epsey, Jones, and Brown).
- Lubricants
  - Report of Committee D-2, 316.

Lumber
  See Wood.

M

Magnesium
  compression testing utilizing rapid heating (Fenn), 940 (*R*). Disc., 955.
Magnesium Alloys
  atmospheric exposure tests
    ASTM test sites (Adam and Dougherty). Report of Committee B-7 (appendix II), 186.
  elevated temperature properties (Voorhees and Freeman) (*Issued as STP 291*).
Magnetic Particle Inspection
  Report of Committee E-7, 471.
Magnetic Properties
  Report of Committee A-6, 124.
Magnetic Testing
  Report of Committee A-6, 124.
Masonry
  brick panels, moisture penetration of (Ritchie and Plewes). Published in ASTM BULLETIN, No. 249, Oct., 39 (TP 183).
  ceramic, potential and past moisture expansion, tests for (Waters, Hosking, and Hueber). Published in ASTM BULLETIN, No. 245, April, 55, (TP 61).
Mass Spectrometry
  Report of Committee E-14, 484.
Materials
  education in
    Report of Administrative Committee on Education in Materials, 86.
Materials Research
  for defense (Speight). Published in ASTM BULLETIN, No. 250, Dec., 6.
Materials Sciences
  published in ASTM BULLETIN, No. 249, Oct., 22; No. 250, Dec., 11.
  Report of Board of Directors, 54.
  See also Symposium on Nature and Origin of Strength of Materials—Summary of Proceedings of *STP 283*, 499.
Measurement
  the meaning of (Baker). Published in ASTM BULLETIN, No. 245, April, 42. (ACR notes).
Medals
  Charles B. Dudley Medal. Published in ASTM BULLETIN No. 247, July, 28.
Meetings
  Annual Meeting—1960 (63rd)
    summary of Proceedings, 1
    Report of Board of Directors, 45
    published in ASTM BULLETIN, No. 244, Feb., 5; 245, April, 7; No. 246, May, 5; No. 247, July, 5.
      awards of merit, No. 247, July, 22.
      committee, technical, new officers for 1960, No. 248, Sept., 20.
      honorary members elected, No. 247, July, 21.
      Marburg lecture, thirty-third (Mark), No. 245, April, 31.
      Marburg and Gillett lecturers, No. 245, April, 10.
      medals and awards, No. 247, July, 28.
      national officers elected, No. 247, July, 18.
      new tentatives and standards, No. 247, July, 31.
      photographic exhibit, 1960 prize winners, No. 248, Sept., 16.
      president's address, international aspects of ASTM (LaQue), No. 247, July, 7.
      preview, No. 243, Jan., 26; No. 244, Feb., 5; No. 246, May, 5.
      provisional program, No. 245, April, 18.
      sessions and symposia. Published in ASTM BULLETIN, No. 247, July, 9.
      testing apparatus exhibit. Published in ASTM BULLETIN, No. 245, April, 11.
  Committee Week (1960)
    near record in Chicago. Published in ASTM BULLETIN, No. 245, April, 45.
    preview, 1961 meeting in Cincinnati, Ohio, Jan. 30–Feb. 3. Published in ASTM BULLETIN, No. 250, Dec., 5.
    Report of Board of Directors, 1960, 45.
  Report of Board of Directors, 40.
  Third Pacific Area National Meeting
    Report of Board of Directors, 45.
Membership
  Report of Board of Directors, 47.
Metal Cleaners
  bibliographical abstracts (Harris) (*issued as STP 90-E*, 1960).
Metal Coatings
  Report of Committee A-5, 111.
  Report of Committee C-22, 295.
Metal Powders
  Report of Committee B-9, 230.
  Report of Committee E-3, 452.
Metallography
  Report of Committee E-4, 457.
  Symposium on Methods of Specimen Preparation. Summary of Proceedings of *STP 285*, 1003:
    controlling surface pitting in polishing ceramic materials (Snow).
    introduction (Norton).
    polishing
      automatic (Anderson).
      mechanical (Link).
      vibratory (Rothstein and Turner).
      vibratory in hot cell application (Long, Meador, and Gray).
    retention and identification of inclusions in steel (Brandenburg).
    techniques for nuclear reactor materials (Cain).
    use of diamond abrasions (Olden).
  the pendulum swings on (Sisco). Published in ASTM BULLETIN, No. 248, Sept., 18. (ACR notes).
Metals
  tensile impact properties of (Steidel and Makerov). Published in ASTM BULLETIN, No. 247, July, 57 (TP 123).
Mica
  Report of Committee D-9, 347.
Microhardness
  tester for high-temperature use (Westbrook).

Published in ASTM BULLETIN, No. 246, May, 53 (TP 113).
Mineral Materials, Sorptive
  Report of Committee C-23, 297.
Missile Materials
  See Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials—Summary of Proceedings of *STP 287*, 1004, *and* Symposium on Acoustical Fatigue—Summary of Proceedings of *STP 284*, 1001.
Moisture
  penetration of brick masonry panels (Ritchie and Plewes). Published in ASTM BULLETIN, No. 249, Oct., 39 (TP 183).
  surface, at corrosion sites, measurement of (Sereda). Published in ASTM BULLETIN, No. 246, May, 47 (TP 107).
Molybdenum
  Report of Committee B-2, 135.
Mortars
  chemical-resistant
    Report of Committee C-3, 253.
  Report of Committee C-1, 247.

N

National Bureau of Standards
  glass research at. Published in ASTM BULLETIN, No. 247, July, 45.
National Officers of ASTM
  elected 1960. Published in ASTM BULLETIN, No. 247, July, 18.
Natural Building Stones
  Report of Committee C-18, 288.
Naval Stores
  Report of Committee D-17, 386.
New Members
  published in ASTM BULLETIN, No. 243, Jan., 86; No. 244, Feb., 70; No. 245, April, 92; No. 246, May, 74; No. 247, July, 88; No. 248, Sept., 66; No. 249, Oct., 66; No. 250, Dec., 70.
Nickel
  Inconel 718 alloy
    mechanical behavior for design of glide vehicle (Tiner and Fischler), 825 (*R*).
  relaxation characteristics of Inconel at elevated temperatures (Kennedy and Douglas), 867 (*R*). Disc., 881.
  Report of Committee B-2, 135.
  Report of Committee E-3, 452.
Nickel Alloys
  Report of Committee B-2, 135.
Nitrogen Resins
  method for urea content, experimental data. Report of Committee D-1 (appendix II), 314.
Nondestructive Testing
  Report of Committee E-7, 471.
Nuclear Fuel
  development (Dalzell) (Gillett Lecture). Published in ASTM BULLETIN, No. 245, April, 10, (*also issued as a separate pamphlet*).
Nuclear Tests
  soils
    See Symposium on Nuclear Methods for Measuring Soil Density and Moisture—Summary of Proceedings of *STP 293*, 1166.

O

Observation
  See Quality of Observation, 1204.
Officers
  national ASTM, elected 1960. Published in ASTM BULLETIN, No. 247, July, 18.
  retired ASTM committee. Published in ASTM BULLETIN, No. 248, Sept., 14.
  technical committee, elected 1960. Published in ASTM BULLETIN, No. 248, Sept., 20.
Oils
  insulating, determination of dielectric strength (Ast). Published in ASTM BULLETIN, No. 250, Dec., 36 (TP 214).
  insulating, development of (Furby and Hanly). Published in ASTM BULLETIN, No. 248, Sept., 41 (TP 155).
Orthophosphate Test
  Report of Committee D-19, 393.
OTS Research Reports
  published in ASTM BULLETIN, No. 244, Feb., 74; No. 245, April, 124; No. 246, May, 72; No. 247, July, 95; No. 248, Sept., 79; No. 249, Oct., 93; No. 250, Dec., 93.
Oxides
  magnesium, manganic, sodium, and potassium, in portland cement, by flame photometry (Ford). Published in ASTM BULLETIN, No. 250, Dec., 25 (TP 203).
Oxidizers
  highly reactive, method to determine impact sensitivity of materials with (Kopituk). Published in ASTM BULLETIN, No. 250, Dec., 51 (TP 229).
Oxygen
  liquid, sensitivity to detonation of materials in contact with, instrument for testing (Lucas and Riehl). Published in ASTM BULLETIN, No. 244, Feb., 29 (TP 33).

P

Packaging
  Report of Committee D-10, 353.
  Report of Committee F-2, 495.
Paint
  latex, permeability to water (Simon). Published in ASTM BULLETIN, No. 249, Oct., 48 (TP 192).
  Report of Committee D-1, 300.
Paper
  Report of Committee D-6, 336.
Paperboard
  Report of Committee D-6, 336.
Papers and Publications
  Report of Administrative Committee on Papers and Publications, 79.
  Report of Board of Directors, 46.
Particle Size
  and dispersion of tungsten powder, effect of dispersion techniques (Michaels, Weaver, and Nelson). Published in ASTM BULLETIN, No. 247, July, 74 (TP 140).

Penetration Tester
  for measuring hardness of polymeric materials (Gardiner, Jordan, and Adams). Published in ASTM BULLETIN, No. 246, May, 38 (TP 98).
Permeability
  plastics, calibration technique for measuring apparatus (McAvoy). Published in ASTM BULLETIN, No. 243, Jan., 52 (TP 10).
  sand stabilized with a chemical grout (Dorion, Burkhard, and White). Published in ASTM BULLETIN, No. 250, Dec., 34 (TP 212).
  to water, of latex paints (Simon). Published in ASTM BULLETIN, No. 249, Oct., 48 (TP 192).
Petroleum
  Report of Committee D-2, 316.
Phenolic
  moldings, flexural and impact strength (Elmer and Harrington). Published in ASTM BULLETIN, No. 249, Oct., 35 (TP 179).
Photographic Exhibit
  ASTM, 1960 prize winners. Published in ASTM BULLETIN, No. 248, Sept., 16.
Photometric Methods of Chemical Analysis
  Report of Committee E-3, 452.
Photometry
  flame, to determine magnesium, manganic, sodium, and potassium oxides in portland cement (Ford). Published in ASTM BULLETIN, No. 250, Dec., 25 (TP 203).
Phthalate Ester Color of High-Gravity Glycerin
  historical review and supporting data for methods. Report of Committee D-1 (appendix I), 311.
Pigments
  lightfastness testing of (Spengeman and Wormald). Published in ASTM BULLETIN, No. 249, Oct., 29 (TP 173).
  Report of Committee D-1, 300.
  Report of Committee D-24, 423.
Pipe
  clay
    Report of Committee C-4, 255.
  concrete, Report of Committee C-13, 275.
  plastic, Report of Committee D-20, 402.
Pitch
  roofing materials
    Report of Committee D-8, 345.
Plaster
  gypsum, normal consistency of, method for determination (Kuntze). Published in ASTM BULLETIN, No. 246, May, 35 (TP 95).
  gypsum, physical changes in setting (Disc.). Published in ASTM BULLETIN, No. 243, Jan., 51 (TP 9).
Plastic Beams
  predicting creep deflections (Schulte), 895.
Plastics
  accelerated aging of vinyl plastic compounds (Royen). Published in ASTM BULLETIN, No. 243, Jan., 43 (TP 1).
  compatibility of magnet wire insulations and epoxy encapsulating resins (Lee). Published in ASTM BULLETIN, No. 247, July, 69 (TP 135).
  hardness, abrasion, and wear resistance, testing (Boor). Published in ASTM BULLETIN, No. 244, Feb., 43 (TP 47).
  penetration tester for measuring hardness of polymeric materials (Gardner, Jordan, and Adams). Published in ASTM BULLETIN, No. 246, May, 38 (TP 98).
  permeability, calibration technique for measuring apparatus (McAvoy). Published in ASTM BULLETIN, No. 243, Jan., 52 (TP 10).
  phenolic moldings, flexural and impact strength (Elmer and Harrington). Published in ASTM BULLETIN, No. 249, Oct., 35 (TP 179).
  polyethylene
    brittleness temperature, test for (Bata and Ito). Published in ASTM BULLETIN, No. 248, Sept., 55 (TP 165).
    compression-molded, creep characteristics of (Gohn and Cummings). Published in ASTM BULLETIN, No. 247, July 64 (TP 130).
    environmental stress-cracking, measurement of (Rudin and Birks). Published in ASTM BULLETIN, No. 245, April, 60 (TP 66).
    measurement of brittleness temperature (Birks and Rudin). Published in ASTM BULLETIN, No. 246, May, 49 (TP 109).
  Report of Committee D-9, 347.
  Report of Committee D-20, 402.
Polyethylene
  brittleness temperature, measurement of (Birks and Rudin). Published in ASTM BULLETIN, No. 246, May, 49 (TP 109).
  brittleness temperature, test for (Bata and Ito). Published in ASTM BULLETIN, No. 248, Sept., 55 (TP 165).
  compression-molded, creep characteristics of (Gohn and Cummings). Published in ASTM BULLETIN, No. 247, July, 64 (TP 130).
  environmental stress-cracking, measurement of (Rudin and Birks). Published in ASTM BULLETIN, No. 245, April, 60 (TP 66).
Polymers
  hardness of, penetration tester for measuring (Gardiner, Jordan, and Adams). Published in ASTM BULLETIN, No. 246, May, 38 (TP 98).
  new,—new problems (Mark) (33rd Marburg lecture). Published in ASTM BULLETIN, No. 245, April, 31.
  See also Plastics.
Porcelain Enamel
  Report of Committee C-22, 295.
Portland Cement
  Report of Committee C-1, 247.
  See also Cement
Precision Data
  proposed glossary of statistical terms. Report of Committee E-15 (appendix), 489.
Preservatives, Wood
  Report of Committee D-7, 339.
President's Address
  international aspects of ASTM (LaQue), 37. Published in ASTM BULLETIN, No. 247, July, 7.

- Pressure Vessel Materials
  - low-cycle fatigue (Sachs, Gerberich, Weiss & Latorre), 512.
- Proposed Glossary
  - of statistical terms. Report of Committee E-15 (appendix), 489.
- Proposed Methods
  - building materials
    - surface flammability. Report of Committee E-5 (appendix), 462.
  - petroleum products
    - autoignition temperature of petroleum products. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1073.
    - automatic digital computer subroutine for volume correction tables. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1069.
    - depentanization of gasolines and naphthas. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1052.
    - effect of air supply on smoke density in distillate fuel burning equipment. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1041.
    - isolation of saturates fraction from low-olefinic stocks. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1056.
    - load-carrying capacity of steam turbine oils. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1083.
    - naphthene in reformer charge and product by refractivity intercept. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1049.
    - particulate matter in aviation fuels. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1077.
    - preparing specimen grids by electron microscopy. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1064.
    - sulfate content of oil-soluble sulfonates. Published in 1960 Compilation of ASTM Standards on Petrolum Products, p. 1061.
    - tetraethyllead in gasoline by flame photometer. Published in 1960 Compilation of ASTM Standards on Petroleum Products, p. 1044.
  - plastics
    - light dosage in light and weathering test apparatus. Published in 1960 Compilation of ASTM Standards on Plastics, p. 595.
  - tex system
    - designation of yarn number, introduction. Published in ASTM BULLETIN, No. 247, July, 48. Also published in 1960 Compilation of ASTM Standards on Textile Materials, p. 818.
  - soaps and detergents
    - analysis of caustic soda by solution A-C arc technique. Published in 1960 Compilation of ASTM Standards on Soaps and Other Detergents, p. 259.
    - determination of solids by infrared moisture balance. Published in 1960 Compilation of ASTM Standards on Soaps and Other Detergents, p. 265.
    - measuring redeposition of soil on cotton fabric. Published in 1960 Compilation of ASTM Standards on Soaps and Other Detergents, p. 266.
- Proposed Recommendations
  - affecting standards
    - copper and copper alloys, cast and wrought. Report of Committee B-5 (appendix), 149.
    - light metals and alloys, cast and wrought. Report of Committee B-7 (appendix I), 174.
    - steel. Report of Committee A-1 (appendix), 93.
    - textile materials. Report of Committee D-13 (appendix), 367.
  - fire testing
    - surface flammability of building materials. Report of Committee E-5 (appendix), 462.
  - precision data
    - proposed glossary of statistical terms. Report of Committee E-15 (appendix), 489.
  - for interlaboratory testing of industrial aromatic hydrocarbons and related materials. Report of Committee D-16 (appendix), 381.
- Protein Materials
  - Report of Committee D-25, 426.
- Publications
  - new ASTM publications. Published in ASTM BULLETIN, No. 243, Jan., 22; No. 244, Feb., 11; No. 245, April, 39; No. 246, May, 8; No. 247, July, 36; No. 248, Sept., 10; No. 249, Oct., 12; No. 250, Dec., 8.
  - Report of Administrative Committee on Papers and Publications, 79.
  - Report of Board of Directors, 46.
  - to be published by ASTM in 1960–1961. Published in ASTM BULLETIN, No. 249, Oct., 12.
- Punch Card
  - handling of atmospheric data (Ailor and Hodgson). Published in ASTM BULLETIN, No. 250, Dec., 45 (TP 223).

Q

- Quality Control
  - Report of Committee E-11, 477.
- Quality of Observation
  - Symposium on Quality of Observation—Summary of Proceedings, 1204:
    - evaluating accuracy (Youden).
    - planned experiments analysis (Terry).
    - precision and accuracy meaning (Murphy).
    - precision; how to evaluate (Conner).
- Quicklime
  - Report of Committee C-7, 257.

R

- Radiant Heating
  - evaluation of thermocouple attachment (Burcham), 957. Disc., 971.

Radiography
  Report of Committee E-7, 471.
Radioisotopes
  Report of Committee E-10, 476.
Random Samples
  current materials news. Published in ASTM BULLETIN, No. 243, Jan., 78; No. 247, July, 52; No. 248, Sept., 32.
Radiation
  solar, use of the Langley for measuring (Caryl). Published in ASTM BULLETIN, No. 243, Jan., 55 (TP 13).
Radiation Effects
  Report of Committee E-10, 476.
  Symposium on Radiation Effects and Radiation Dosimetry—Summary of Proceedings of *STP 286*, 1203:
    Bragg-Gray cavity chamber (Ritz and Attix).
    effect of neutron irradiation on Charpy and drop weight test (Hawthorne and Steele).
    energy absorption
      effect of sample parameters (Johnston).
    fuel depletion studies
      radiochemistry and mass spectrometry (Hudgens).
    gamma ray dosimetry (Hale, Johnson, Dec, Coss, Sessoms, Hemmig, Rice, and Brocklehurst).
    irradiation testing in CP-5 reactor (Silliman, Shoudy, Huebotter, and Blessing).
    materials irradiation studies (Steele and Hawthorne).
    measurement of energy absorbed from pile neutrons (Binder, Bopp, and Towns).
    neutron spectrum determinations at low flux levels (Kruger).
    parameters in testing high polymers (Fritz).
    reactor spectra consideration (Romanko).
Refractories
  Report of Committee C-8, 260.
Refractory Metals
  high-temperature, vacuum equipment for treatment of (Field and Peckham). Published in ASTM BULLETIN, No. 244, Feb., 34 (TP 38).
  in superalloys (Parker), 849 (*R*). Disc., 866.
  tungsten powder, particle size and distribution (Michaels, Weaver, and Nelson). Published in ASTM BULLETIN, No. 247, July, 74 (TP 140).
Research
  Report of Administrative Committee on Research, 78.
Resins
  epoxy, encapsulating, compatibility with magnet wire insulations (Lee). Published in ASTM BULLETIN, No. 247, July, 69 (TP 135).
  nitrogen
    method for urea content, experimental data. Report of Committee D-1 (appendix II), 314.
  solutions
    viscosity, interlaboratory data. Report of Committee D-1 (appendix III), 315.
Resistance Strain Gages
  evaluation at elevated temperatures (Bloss), 1207.
Rheology
  of elastomers, at low temperatures (Boor, Hanok, Conant, and Scoville). Published in ASTM BULLETIN, No. 246, May, 25 (TP 85).
Road and Paving Materials
  Session. Summary of Proceedings of *STP 294*, 1167:
    air-permeabilities of asphalt concrete (Ellis and Schmidt).
    effect of compaction temperature, bituminous concrete (Kiefer).
    emulsified asphalt tests (McConnaughay).
    influence of asphalt composition on Rheology (Winniford).
    laboratory tests and specifications for setting and mixing (Mertens, Coyne, and Rogers).
    stress deformation characteristics of sand bituminous mixtures (Carpenter).
Road Materials
  Report of Committee D-4, 328.
  Report of Committee D-18, 388.
Roofing Materials
  Report of Committee D-8, 345.
Rosin
  Report of Committee D-17, 386.
Roughness Factor
  of small steel disk, measurement of (Begovich). Published in ASTM BULLTEIN, No. 248, Sept., 56 (TP 166).
Rubber
  and other elastomeric vulcanizates, method for classifying (Catton, Edwards, and Loring). Published in ASTM BULLETIN, No. 243, Jan., 69 (TP 27).
  Report of Committee D-11, 355.
Rubber Chemicals
  Report of Committee D-11, 355.
  Report of Committee D-24, 423.

S

Sand
  permeability of, stabilized with chemical grout (Dorion, Burkhard, and White). Published in ASTM BULLETIN, No. 250, Dec., 34 (TP 212).
  relative density of, measurement (Kitago and Kozaki). Published in ASTM BULLETIN, No. 248, Sept., 36 (TP 150).
  standard Ottawa, test method for air-entrainment of (DeFore and Corah). Published in ASTM BULLETIN, No. 248, Sept., 48 (TP 162).
Sandwich Construction
  peel test
    Report of Committee D-14, 371.
Science
  improving, in secondary education (Shobert). Published in ASTM BULLETIN, No. 246, May, 7 (ACR notes).
Sealants
  for joints
    Report of Committee C-24, 298.

Semiconductors
  Report of Committee F-1, 491.
Shear and Torsion Testing
  Symposium. Summary of Proceedings of *STP 289*, 1002:
    adhesive bonding—preparation of metal surface (Shane).
    load transfer in weld spots (Freytag).
    metal-to-metal bond (Lunsford).
    metal-to-metal bond—lap joint shear test (Shane).
    panel shear test
      photoelastic evaluation (Bryan).
    photoelastic coating (Zandman).
    refractory materials at 2800 C (Waller and Stehsel).
    solid materials (Clapper).
    stress distribution (Yen).
    study of high polymers by torsion pendulum (McCrum).
    tensile loaded specimen design and development (Zapel).
    test for determining shear modulus of sandwich core materials (Raville, Bickford, and Huber).
    wood, plywood, and sandwich constructions (Youngquist and Kuenzi).
Sheet Materials
  high-strength, fracture testing of (report of special ASTM committee). Published in ASTM BULLETIN, No. 243, Jan., 29; No. 244, Feb., 18.
Sheets, Iron and Steel
  Report of Committee A-5, 111.
Shipping Containers
  Report of Committee D-10, 353.
Siding Materials
  Report of Committee D-8, 345.
Silicon Steel
  Report of Committee A-6, 124.
Simulated Service Testing
  Report of Administrative Committee (appendix VI), 85.
Soaps
  Report of Committee D-12, 361.
Sodium
  movement of, in portland cement (Hall and Rhodes). Published in ASTM BULLETIN, No. 245, April, 66 (TP 72).
  Report of Committee B-2, 135.
Soils
  frost action
    importance of freezing rate (Penner), 1151 (*R*).
  preloaded fine grained
    settlement (Zegarra), 1109, Disc., 1118.
  remolded Niagara clay
    compressive strength (Bazett and Smotrych), 1124. Disc., 1132.
  Report of Committee D-18, 388.
  stress-strain relations
    duplication of prototype (Ahlvin and Brown), 1137, Disc., 1149.
  Symposium on Nuclear Methods for Measuring Soil Density and Moisture—Summary of Proceedings of *STP 293*, 1166:
    airfield pavement construction (Carlton).
    evaluating equipment (Carey, Shook, and Reynolds).
    neutron Moisture meter (Burn).
    nuclear Chicago d/M gage (Neville and Van Zelst).
    sand cone methods (Mintzer).
    surface probes on O'Hare Field (Gnaedinger).
Solar energy
  utilization (Daniels) (Marburg Lecture) Published in ASTM BULLETIN No. 245, April, 10 (*also issued as a separate pamphlet*).
Solder
  Report of Committee B-2, 135.
Solvents
  halogenated organic
    Report of Committee D-26, 428.
  Report of Committee D-1, 300.
Sorptive Mineral Materials
  Report of Committee C-23, 297.
Spectrometry
  Mass
    Report of Committee E-14, 484.
Spectroscopy
  Report of Committee E-2, 448.
  Report of Committee E-13, 482.
  Report of Committee E-14, 484.
  X-Ray
    Index to the Literature, Part I, 1913–1957. (*issued as STP 292*).
Spin Fracture Test
  rotor steels in brittle fracture (Sankey), 721 (*R*). Disc., 733.
Standardization
  actions on standards. Published in ASTM BULLETIN, No. 243, Jan., 25; No. 244, Feb., 28; No. 245, April, 30; No. 246, May, 10; No. 247, July, 31; No. 250, Dec., 10.
  and the purchaser (Brunberg). Published in ASTM BULLETIN, No. 249, Oct., 6.
  federal government standards changes. Published in ASTM BULLETIN, No. 243, Jan., 102; No. 244, Feb., 89; No. 245, April, 125; No. 247, July, 112; No. 248, Sept., 96; No. 249, Oct., 91; No. 250, Dec., 94.
  larger U. S. effort in international (Scott). Published in ASTM BULLETIN, No. 249, Oct., 10.
  Report of Administrative Committee on Standards, 71.
Standards
  ASTM, for highway materials (Hveem). Published in ASTM BULLETIN, No. 249, Oct., 8.
  sound, stimulate manufacturing (McCoy). Published in ASTM BULLETIN, No. 248, Sept., 5.
Static and Fatigue Strength
  metals subjected to triaxial stresses (Lehrer and Schwartzbart), 610 (*R*).
Statistical Analysis
  Report of Committee E-11, 477.
Statistical Terms
  glossary. Report of Committee E-15 (appendix), 489.

- Steel
  - austenitic chromium-nickel stainless
    - effect of alloying additions on hot cracking (Hull), 667 (*R*). Disc., 686.
  - carbon and low alloy
    - creep rupture properties at 1200 to 1800 F (Randall), 885. Disc., 893.
  - corrosion
    - Report of Advisory Committee on Corrosion, 234.
    - Report of Committee A-5, 111.
    - Report of Subcommittee XVI of Committee, A-5, 116.
  - corrosion-resisting
    - Report of Committee A-10, 126.
  - effect of carbon content and melting practice (Shannon, Epsey, Repko, and Brown), 761 (*R*). Disc., 778.
  - fatigue of high-strength steels (Frankel, Bennett, and Carman), 501, Disc., 509.
  - high-strength
    - delayed failure in liquid environments (Steigerwald), 750.
  - low alloy
    - atmospheric corrosion (Copson), 650 (*R*). Disc., 666.
  - Report of Committee A-1, 88.
  - Report of Committee A-6, 124.
  - Report of Committee A-10, 126.
  - Report of Committee E-3, 452.
  - rotor
    - spin fracture tests (Sankey), 721 (*R*). Disc., 733.
  - SAE 4340, low-temperature tensile-hardness correlations (Nunes and Larson). Published in ASTM Bulletin, No. 249, Oct., 25 (TP 169).
  - sheets
    - Report of Committee A-5, 111.
  - stainless
    - temperature dependence of the elastic moduli (Garofalo), 738 (*R*).
  - structural alloys
    - Strength under rapid heating and loading (Kattus and McDowell), 928.
  - ultra-high-strength
    - carbon:strength relationships (Hamaker and Vater), 691 (*R*). Disc., 717.
  - *See also* Proposed Recommendations.
- Stone
  - building
    - Report of Committee C-18, 288.
- Straightness
  - of fine wire, apparatus for measuring (Nelson and Rhodes). Published in ASTM Bulletin, No. 244, Feb., 56 (TP 60).
- Stress-Cracking
  - environmental, of polyethylene, measurement of (Rudin and Birks). Published in ASTM Bulletin, No. 245, April, 60 (TP 66).
- Stress Relaxation (Freudenthal), 986 (*R*). Disc., 1000.
- Stress-Rupture
  - high-temperature correlating parameters (Clauss), 905 (*R*).
- Strontium Oxide
  - in portland cement, gravimetric determination of (Ford). Published in ASTM Bulletin, No. 245, April, 71 (TP 77).
- Sulfur Dioxide
  - at atmospheric corrosion sites, measurement of (Sereda). Published in ASTM Bulletin, No. 246, May, 47, (TP 107).
- Super-Strength Alloys
  - Report of Committee A-10, 126.
- Superalloys
  - role of refractory metals (Parker), 849 (*R*). Disc., 866.

T

- Tall Oil
  - Report of Committee D-17, 386.
- Tantalum
  - Report of Committee B-2, 135.
- Tar
  - Report of Committee D-4, 328.
  - Report of Committee D-8, 345.
- Technical Activities
  - Report of Board of Directors, 41.
- Technical Committee Notes
  - Published in ASTM Bulletin, No. 243, Jan., 41; No. 244, Feb., 17; No. 245, April, 47; No. 246, May, 21; No. 247, July, 41; No. 248, Sept., 22; No. 249, Oct., 24; No. 250, Dec., 20.
- Temperature Effect
  - concretes (Zoldners), 1087 (*R*).
  - elastic moduli of stainless steel (Garofalo), 738 (*R*).
  - on properties of metals. Report of the Joint ASTM-ASME Committee on Temperature Effects on the Properties of Metals, 241.
  - Report of Committee B-4, 143.
- Tensile Strength
  - SAE 4340 steel, low-temperature, correlation with hardness (Nunes and Larson). Published in ASTM Bulletin, No. 249, Oct., 25 (TP 169).
- Tension
  - phosphor bronze rod, alloy A (Gohn and Bader), 843.
  - short-time elevated temperature
    - application of induction heating (Levitt and Martin), 974. Disc., 984.
  - steel
    - effect of carbon content and melting practice (Shannon, Epsey, Repko, and Brown), 761 (*R*). Disc., 778.
- Tension Testing
  - elevated temperature
    - unalloyed arc-cast molybdenum (Brock and Sinclair), 530. Disc., 538.
  - impact properties of metals (Steidel and Makerov). Published in ASTM Bulletin, No. 247, July, 57 (TP 123).
  - machining of notched specimens for (March, Ruprecht, and Reed). Published in ASTM Bulletin, No. 244, Feb., 52 (TP 56).
- Testing Machines
  - Report of Committee E-1, 439.

Tetraethyllead
  determination in gasoline. Published in ASTM BULLETIN, No. 250, Dec., 13.
Tex System
  designation of yarn number, introduction of. Published in ASTM BULLETIN, No. 247, July, 48.
Textile Materials
  Report of Committee D-13, 365.
  See also Proposed Recommendations.
Thermal Insulating Materials
  Report of Committee C-16, 281.
Thermal Stability
  of disiloxane-base hydraulic fluids, determination of (Willard). Published in ASTM BULLETIN, No. 249, Oct., 52 (Tp 196).
Thermocouples
  attachment methods for radiant heating applications (Burcham), 957, Disc. 971
  Report of Committee E-1, 439.
Thermometers
  Report of Committee E-1, 439.
Thermostat Metals
  Report of Committee B-4, 143.
Thorium
  Report of Committee B-2, 135.
Tile
  clay
    Report of Committee C-15, 277.
  facing
    Report of Committee C-15, 277.
Timber
  Report of Committee D-7, 339.
  See also Wood.
Tin
  Report of Committee B-2, 135.
Titanium
  design criteria for 6Al-4V alloy (Childs and Lemcoe). Published in ASTM BULLETIN, No. 246, May, 41 (TP 101).
  Report of Committee B-2, 135.
  Report of Committee E-3, 452.
Transformer Oil
  Report of Committee D-27, 429.
Tungsten
  powder, particle size and distribution, effect of dispersion techniques (Michaels, Weaver, and Nelson). Published in ASTM BULLETIN, No. 247, July, 74 (TP 140).
  Report of Committee B-2, 135.

U

Ultrasonic Testing
  Report of Committee E-7, 471.
Uranium
  Report of Committee B-2, 135.

V

Vacuum
  equipment for high-temperature treatment of reactive and refractory metals (Feild and Peckham). Published in ASTM BULLETIN, No. 244, Feb., 34 (TP 38).
  fatigue testing machine, high-temperature axial (Stephens and Sinclair). Published in ASTM BULLETIN, No. 249, Oct., 44 (TP 188).
Varnish
  insulating
    Report of Committee D-9, 347.
  Report of Committee D-1, 300.
Vinyl
  plastics, accelerated aging of, (Royen). Published in ASTM BULLETIN, No. 243, Jan., 43 (TP 1).
Viscometer
  capillary, alignment block and clamp for (Robinson). Published in ASTM BULLETIN, No. 245, April, 69 (TP 75).
  modified MacMichael, to determine consistency of printing inks and ink vehicles (Cummings). Published in ASTM BULLETIN, No. 250, Dec., 49 (TP 227).
Viscosity
  cellulose
    Report of Committee D-23, 420.
  resin solutions
    interlaboratory data. Report of Committee D-1 (appendix III), 315.

W

Water
  industrial
    Manual on Industrial Water and Industrial Waste Water (*issued as STP 148-E, 1960*).
  old wine in new bottles, a new look at the substance, water (Clarke). Published in ASTM BULLETIN, No. 243, Jan., 17.
  Report of Committee D-19, 393.
Waterproofing
  Report of Committee D-8, 345.
Wax
  Report of Committee D-2, 316.
  Report of Committee D-21, 415.
Wear
  carbon brushes, in an atmosphere containing silicone vapor, technique for measuring (Axelson and Precopio). Published in ASTM BULLETIN, No. 244, Feb., 40 (TP 44).
  resistance of plastics, testing (Boor). Published in ASTM BULLETIN, No. 244, Feb., 43 (TP 47).
Weatherability
  coating grade asphalts, prediction from asphaltene characteristics (Mertens). Published in ASTM BULLETIN, No. 250, Dec., 40 (TP 218).
Whitewares
  Report of Committee C-21, 291.
Wire
  copper
    Report of Committee B-1, 130.
  electrical conductors
    Report of Committee B-1, 130.
  insulating
    Report of Committee D-11, 355.
  straightness of, apparatus for measuring (Nelson and Rhodes). Published in ASTM BULLETIN, No. 244, Feb., 56 (TP 60).

Wire (*continued*)
  zinc-coated and uncoated
    twenty-year atmospheric corrosion (Reinhart) (*issued as STP 290*).
Wood
  Report of Committee C-7, 257.
Wood-Base Materials
  Report of Committee D-7, 339.
Wood Poles
  Report on Strength and Related Properties (*issued as STP 295*).
Wood Preservatives
  Report of Committee D-7, 339.
Writing
  technical, the labor of (Smith). Published in ASTM BULLETIN, No. 250, Dec., 12. (ACR notes).

X

X-Ray Diffraction
  hydrated portland cement pastes (Kantro, Copeland, and Anderson), 1020 (*R*). Disc., 1035.

Y

Yarn Number
  introduction of the tex system, for designating. Published in ASTM BULLETIN, No. 247, July, 48.
Yarns
  Report of Committee D-13, 365.

Z

Zinc
  alloy test bars
    mechanical properties. Report of Committee B-6 (appendix I), 162.
  corrosion
    of coated hardware. Report of Subcommittee XVI of Committee A-5, 116.
  Report of Committee B-2, 135.
  Report of Committee B-6, 160.
Zinc Coated Wire
  twenty-year atmospheric corrosion (Reinhart) (*issued as STP 290*).
Zirconium
  Report of Committee B-2, 135.
  Report of Committee E-3, 452.

1960 LIST OF SPECIAL TECHNICAL PUBLICATIONS

The publications listed below have been compiled during the year 1960 to be issued as separate publications not included in either *Proceedings* or ASTM BULLETIN. For a list of earlier Special Technical Publications, see the original listings on the last page of *Proceedings*, Vol. 49, (1949) to Vol. 59 (1959). Also a complete list of Special Technical Publications covering all publications, up to and including 1950, is included in the 50-Year Index to ASTM Publications, and a complete list up to and including 1955 is published in the 5-Year Index to ASTM Publications (1951–1955).

SPECIAL TECHNICAL PUBLICATION NUMBER	TITLE
48-H	X-ray Diffraction Data Cards for Chemical Analysis, with Alphabetical and Numerical Indexes. Yearly Supplement—Section 9 (1959).
48-I	Yearly Supplement—Section 10 (1960).
48-J	Revised Sections 1 to 5 (1960).
56-N	1959 Supplement to the Bibliography and Abstracts on Electrical Contacts (1960).
58-D	Report on Standard Samples and Related Materials for Spectrochemical Analysis (1960).
90-E	1960 Supplement to the Metal Cleaning Bibliography (1960).
148-E	Manual on Industrial Water and Industrial Waste Water (1960).
150-C	1959 Supplement to Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (1960).
170-B	Compilation of Chemical Composition and Rupture Strengths of Super-Strength Alloys (1960).
*266	Symposium on Effect of Water-Reducing Admixtures and Set-Retarding Admixtures on Properties of Concrete (1959).
*267	Symposium on Hydraulic Fluids (1959).
*268	Symposium on Applied Radiation and Radioisotope Test Methods (1959).
*269	Symposium on Spectroscopy (1959).
*270	Symposium on Durability and Weathering of Structural Sandwich Constructions (1959).
*271	Symposium on Adhesion and Adhesives (1959).
*272	Symposium on Newer Metals (1959).
*273	Symposium on Technical Developments in the Handling and Utilization of Water and Industrial Waste Water (1959).
*274	Symposium on Fatigue of Aircraft Structures (1959).
*275	Symposium on Treated Wood for Marine Use (1959).
*276	Materials in Nuclear Application (1959). Symposium on Ceramics in Nuclear Energy Symposium on Radiation Effects and Dosimetry Symposium on Post Irradiation Effects in Polymers
*277	Symposium on Road and Paving Materials (1959).
*278	Symposium on Nondestructive Testing in the Missile Industry (1959).
*279	Reinforced Plastics for Rockets and Aircraft (1959). Symposium on Reinforced Plastics Symposium on Thermal Ablation
*280	Symposium on Bituminous Waterproofing and Roofing Materials (1959).
*281	Symposium on Air Pollution Control (1959).
*282	Papers on Building Constructions (1959). Symposium on Wood in Building Constructions Symposium on Methods of Testing Building Constructions
283	Properties of Crystalline Solids (1960). Symposium on Recent Progress in Materials Sciences Symposium on Nature and Origin of Strength of Materials
284	Symposium on Acoustical Fatigue (1960).
285	Symposium on Methods of Metallographic Specimen Preparation (1960).
286	Symposium on Radiation Effects and Radiation Dosimetry (1960).
287	Symposium on Low-Temperature Properties of High-Strength Aircraft and Missile Materials (1960)
288	*Unassigned.*
289	Symposium on Shear and Torsion Testing (1960).
290	20-Year Atmospheric Corrosion of Zinc-Coated Wire and Wire Products (1960).
291	Report on the Elevated-Temperature Properties of Aluminum and Magnesium Alloys (1960).
292	Index to the Literature on X-ray Spectrographic Analysis—Part I, 1913–1957 (1960).
293	Symposium on Nuclear Methods for Measuring Soil Density and Moisture (1960).
294	Papers on Road and Paving Materials (Bituminous)—1960.
295	Strength and Related Properties of Wood Poles (1960).

---

* West Coast Papers.